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AMERICAN
DYESTUFF REPORTER

VOLUME 41 September 15, 1952 NUMBER 19

ANALYSIS OF DYES FOR MAN-MADE FIBERS

ALFRED F SCHNEID
Research Laboratories, Interchemical Corporation

ONE of the most rapidly advancing fields of chemistry for the past 100 years has been that of dyestuffs. Ever since the invention of mauve by W H Perkin in 1856, the dyestuff field has advanced by waves of renewed activity. These advances stemmed from the discovery of the diazo reaction by Peter Griess in 1858, the identification of alizarin and its synthesis in 1869, and the synthesis of indigo in 1878. By 1900, tremendous research efforts had introduced hundreds of different intermediates into the rapidly expanding industry. The industry became internationally competitive. A great deal of the total research effort was devoted to finding out what the other fellow had to offer and how it could be bettered.

The only limitations to the development of different dyestuff structures were those imposed by the nature of the fibers to which the dyes could be affixed. The discovery of new synthetic fibers increased the potentiality of new dye structures.

Along with the increasing complexity of synthetic dyes came the urge to develop tests for their analysis. Keeping tabs on the competitor meant long hours of work by resourceful and ingenious analytical chemists. It would take volumes to describe all the systematic procedures advocated for these analyses. Most of this work was done on dyes to be applied to cotton, wool, and silk.

Many good systematic procedures (including some in need of modernization) for analysis of dyes for natural fibers are available in a number of standard dyestuff "bibles" (*3, 4, 8, 12*). This paper will be devoted largely to the analysis of dyestuffs for man-made fibers, such dyes having received relatively little attention up to now. More specifically, the purpose of this article is to present a suitable procedure to be followed by an organic chemist who wishes to determine the structure of an unknown dye for fibers such as acetate and nylon. With this in mind, the procedures outlined have been kept as simple as possible.

A F Schneid

This article presents an outline of procedures to be followed for the analysis of a dye for acetate, nylon, or other man-made fibers of unknown composition. Reprinted from *Interchemical Review, Volume 11,* pages 31-40, 48-52, Summer, 1952. Copyright 1952 by Interchemical Corporation.

Cellulose acetate fibers possess little or no affinity for the great majority of dyes used in the coloring of other fibers. The wetting and penetrating of cellulose acetate is not easy; it becomes still more difficult when the acetyl value is high. For the most part, acetate dyes are insoluble azo or anthraquinone dyes that have been highly dispersed to make them capable of penetrating and dyeing acetate textile fibers. A second class is insoluble amine azo dyes that are made water-soluble by treatment with formaldehyde and bisulfite. After sorption by the fiber, the resulting sulfonic acids hydrolyze and regenerate the insoluble dyes.

Most "general practitioners" of organic chemistry will be content to consider the dyeing of acetate and nylon fibers as a process in which solute, the dyestuff, dissolves in solvent, the fiber. Therefore, any chemical structure which will tend to give the solute solubility properties different from those of the solvent will result in loss of affinity for the fiber. This loss would stem from an increase in water solubility at the expense of organic solubility because of competition for the dye between the dye bath and the fiber. The greater the influence of the solubilizing groups is, the lower is the affinity for the acetate or nylon fiber.

How systematic an analytical procedure may be followed in a particular case will be dependent on a number of factors:

1. The experimental knowledge and powers of observation of the investigator;
2. The physical and chemical nature of the dyestuff whose structure is being determined;
3. The tools the analyst has at his disposal.

Although the first and older dyestuff analysts had the advantage of dealing with fewer possible structures, they deserve to be greatly admired by us moderns for the work they were able to do despite their handicaps. They were limited by lack of instrumentation, as well as by lack of chemical knowledge, a knowledge which we have today in our libraries and chemical publications.

The better the tools, the easier the analytical job will be. The proper use of instruments such as the spectrophotometer or the X-ray diffraction camera may cut the analysis time in half. The use of various types of instruments will be approached in this article from the standpoint of specific applications.

PHYSICAL ANALYSIS

The first thing the investigator should do upon receiving a sample for chemical analysis is to examine the product physically. The label on the bottle alone may suggest something about the consitution of the dye. An acetate dye, for instance, can

be expected to be free of water-solubilizing groups, for, as stated above, such groups lower the affinity of the dyestuffs for acetate or nylon substrates.

The color of the dyestuff can also tell us something about its composition. The fact that the dyestuff being investigated is a red, for example, will give the experienced analyst an insight as to its constitution; combinations of chromophoric and auxochromic groups would be different in a red from those present in a blue or yellow.

It is important for the investigator to know whether or not the unknown is a single dyestuff cut to a definite strength or whether it is a mixture of two or more dyes. Tests to determine this can be made either before or after the pure dyestuff has been extracted from its cutting agent The latter is usually a common dyestuff-dispersing agent such as a sodium lignin sulfonate, dextrin, sugar, alkali phosphate, or even plain salt. These dispersing agents are always water-soluble.

To test whether the unknown is a mixture of two or more dyes, the simple blow-out test will sometimes be effective. This consists in distributing a sample of the finely dispersed dye over a piece of white filter paper moistened with solvent. After this distribution, either from a razor's edge or by blowing, the paper is watched for the spreading of bands of different colors. The test is usually most effective if the solvent used to saturate the paper is a mixture of, for example, an alcohol, a ketone, and a little of an aromatic, such as toluene. This crude test is effective only if the components in the mixture are sufficiently different in color or possess marked differences in solubility in the solvent with which the filter paper has been moistened.

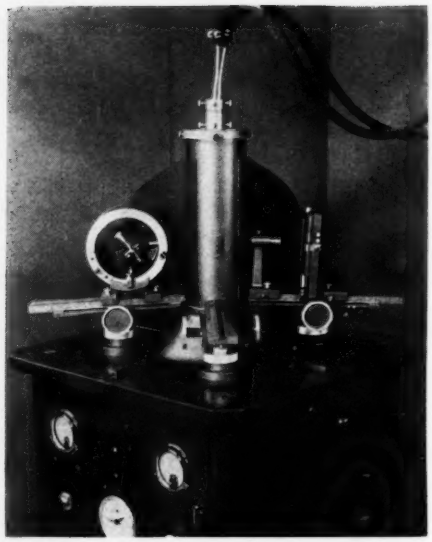
PURIFYING THE DYE

One of the surest ways to detect and even to separate a mixture is by chromatographic adsorption. Since this is best done on a sample free from cutting agents, its theory will be discussed after some recommended procedures are given for extracting the pure dye from its dispersing agents. If the crystalline dyestuff has a high specific gravity or is of high molecular weight, the dispersing agents may sometimes be effectively removed by subjecting a water suspension of the dye to high-speed centrifuging, decanting the liquor which contains the dispersing agent and repeating the operation a number of times. The relatively pure product is then recrystallized or subjected to chromatographic analysis and tested for uniformity. Sometimes the dyestuff can be extracted with a solvent such as acetone in a Soxhlet extraction apparatus. The extract is then drowned in water, filtered, washed, dried, and recrystallized from a suitable solvent.

It may be possible to purify a given unknown dye by sublimation. If this were the case, we should be doubly thankful, for not only would the dye be obtained in a pure crystalline state, but it would reveal something about its basic structure.

A simple sublimation test can be made directly on the melting point block, a very handy tool. A few milligrams of the sample are merely placed on a cover glass on the heated block, and a cover glass is then brought down as close as possible, but not touching the sample. Any vapor from the sample will condense on the cooler upper cover glass in a fairly pure form. The latter can then be inverted, covered, and pressed firm with still another cover glass and the melting point of the collected product can be recorded. A test as simple as this can be done in a matter of minutes and will tell the investigator much about his product.

The melting point block, properly used, has definite advantages over capillary methods. The sample is more easily observed, and color changes are more readily apparent. Resolidification and crystal formation can also be observed more clearly on the block. Much less of a single sample is needed and more samples, representing three or four recrystallizations, for example, can be observed at one time. Simultaneous observations of the melting points of two samples and their mixture are possible. The block is also faster and much safer since there is no danger of spilling hot oil or sulfuric acid. For some cases, however, such as extremely high-melting substances which have a tendency to vaporize from between the two cover glasses before the melting point is reached, the sealed capillary has a distinct advantage. The capillary method is more accurate in such cases because it is less liable to too rapid heating.



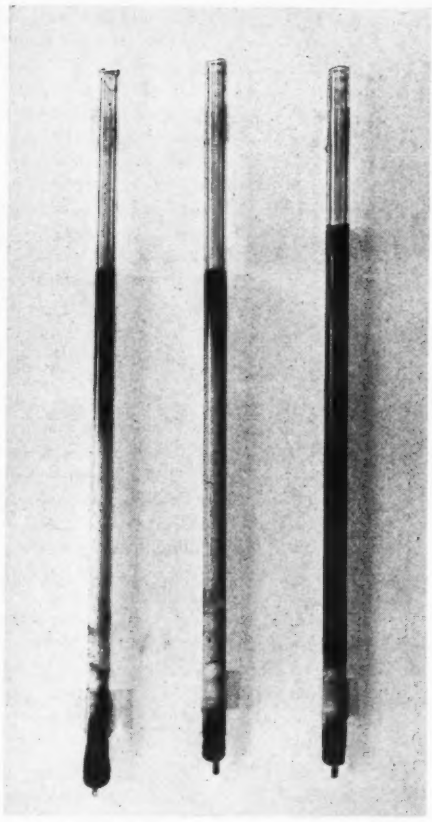
X-Ray Diffraction Unit

CHROMATOGRAPHIC ADSORPTION

The chromatograph, already mentioned as a means of separating and detecting mixtures of dyes, deserves a brief discussion. In principle, chromatographic analysis comprises separation techniques based on differential partition of solutes between a moving and a stationary phase. Chromatography combines adsorption from solution and partition between solvents by such a modification of experimental techniques that adsorption, desorption, and partition are repeated many times. Chromatography is therefore comparable to fractional distillation or fractional crystallization. One of its greatest advantages is that it can be applied to micro quantities of a substance and that often the starting material can be recovered almost quantitatively.

The adsorbents used in a column may be of many different types, since they vary one from another in selectivity and degree of activity. Silica gel, for example, is very selective for primary amino groups, while alumina will adsorb any compound having an hydroxyl group. Frequently the adsorbent is too reactive so that everything is adsorbed and no clear-cut separation is obtained. Alumina especially varies widely in activity.

The nature of the solvent or eluant is very important to the success of a chromatographic analysis (*1*). The competition between solvent and solute for the various points on the solid adsorbent and the distribution coefficients between immiscible solvents largely determine the effectiveness of a particular separation. For hydrophilic adsorbents the order of solvents in the direction of increasing eluting power is:



A dye adsorbed on deactivated (L_x) and activated alumina, but only slightly adsorbed on silica gel column (R_x).

hydrocarbons (e g petroleum ether, benzene); chloroform; diethyl ether; ethyl acetate; acetone; propyl alcohol; then, ethyl and methyl alcohol; and finally, pyridine. This order runs parallel to the dielectric constants of the liquids (*1*).

One can readily establish a relative scale of activity of adsorbents by preparing various samples of aluminum oxide, possessing different degrees of activity (*18*). By employing test solutions consisting of a group of azo dyes which possess different functional groups, the degree of activity can be established rapidly; observations are made of the behavior of different adsorbents under conditions of uniform adsorption (*2*). The dyestuffs chosen are all derivatives of aromatic azo compounds—*p*-hydroxyazobenzene, *p*-aminoazobenzene, Sudan red, Sudan yellow, *p*-methoxyazobenzene, and azobenzene. Table I shows the separation on aluminum oxides with five degrees of activity. Other adsorbents, like silica gel, magnesium oxide and calcium sulfate, can be ranked in comparison to alumina on a similar scale by making use of the same dyes (2). Adsorption activity decreases in the order of oxides, sulfates, carbonates and oxalates. It must always be remembered that the order of adsorption is not only a function of the adsorbent but also of the solvent medium.

The chromatograph, in addition to its successful use for the identification and separation of a mixture, is extremely handy in determining the purity of a synthesized component. Chromatographic techniques also afford an excellent means of purification of synthetic products found to be impure. Very often, particularly for the investigator who is pressed for time and who has had no experience in chromatographic analysis, selective crystallization from solvents is an "old reliable," as techniques go, for separating a mixture. The chief difficulty in its use is that in dyestuff analysis the components of the mixture usually have similar physical characteristics. For example, two components selected for their equal affinity for the fiber so as to insure a level and even dyeing, would probably have similar chemical structures. Therefore, selective crystallization could prove tedious, awkward, and might require large amounts of sample.

Should the investigator already know the structure of one of the components in a mixture and wish to separate the unknown, a somewhat different method can be employed. By chemical reaction such as acetylation or methylation of the known component possessing a reactive group (hydroxyl, amino, etc.) the character of this component can be changed to the degree necessary for its separation from the unknown by fractional or selective crystallization. If such a procedure is used, the unknown should be restored to its original form (e g subjected to hydrolysis if acetylation was used) before identification. In most cases, however, alternative methods eliminate the need for such an elaborate procedure.

A technique often forgotten for separating mixtures, especially of acetate dyes, depends on the difference in solubilities of various organic substances in solutions of different pH. A given mixture, for example, contains two simple azo dyes, one possessing a phenolic hydroxyl group and the other a sulfonamide group. Both are soluble in hot sodium carbonate solutions since both have available replaceable hydrogens, but the addition of sodium bicarbonate will result in the precipitation of the dye having the sulfonamide group. Dyes made from secondary amines can likewise usually be separated from those made from primary amines simply by use of acid solutions, without resorting to the more complex Hinsberg procedure. The simple solubility tests used for the identification of organic compounds often prove applicable after the pure dye has been extracted from its cutting dilutents.

OTHER PHYSICAL TESTS

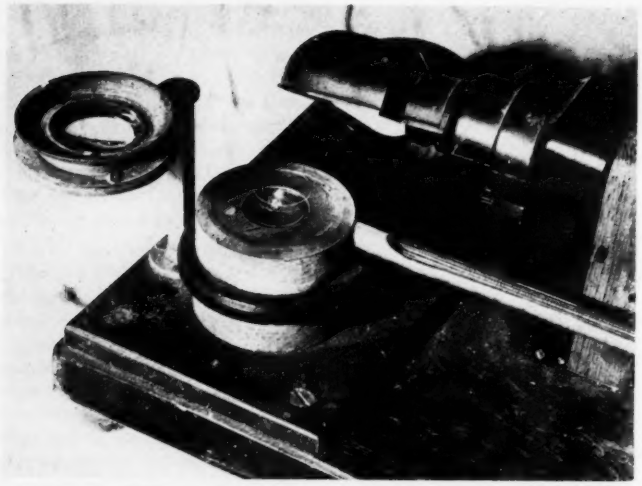
Spectrophotometric curves of the pure

TABLE I

CLASSIFICATION OF ALUMINUM OXIDE ADSORBENTS BY THEIR ACTIVITY IN SEPARATING A SERIES OF DYESTUFFS

Degree of activity	I	II		III		IV		V
		a	b	a	b	a	b	
Location of dye								
Above in column	PMAB		SY		SR			
Below in column	AB		PMAB		SY			
In column		PMAB		SY		SR	PAAB	PHAB
In filtrate		AB		PMAB		SY	SR	PAAB

Key to dyes: AB=azobenzene; PAAB=*p*-aminoazobenzene; PHAB=*p*-hydroxyazobenzene; PMAB=*p*-methoxyazobenzene; SR=Sudan red (*Merck Index:* Sudan III); SY=Sudan yellow (aniline coupled into *Beta*-Naphthol).



Melting point block with magnifying lens turned to show cover glasses with original material and sublimed crystals; thermometer is at lower right.

dye should be made after it has been recrystallized repeatedly to a constant melting point. This "fingerprinting" of the dye, either by visible light or infra-red (or preferably both), yields a permanent comparison record, which may serve as a proof of structure once the dye has been synthesized. The infra-red spectogram is extremely valuable since functional groups such as halogen, hydroxyl, amino, etc., all possess characteristic absorption bands in the infrared region (*29*). For example, all compounds having a free hydroxyl group have a common absorption band at about 2.9 μ. If the hydroxyl is converted to a carbonyl group, the band at 2.9 μ will disappear and a new absorption band will be visible in esters and other compounds with carbonyl groups at approximately 5.8 μ. The characteristic absorption band of the nitrile group is at 4.45 μ. New acetate dyes containing nitrile groups could therefore be spotted (*29*). Where substituent groups with interfering absorption bands are present, slight changes in the molecule may sometimes be effected to remove the interference (*11*).

The infra-red spectrophotometer is also an excellent instrument for determining the purity of a given compound. In many cases it may even enable the investigator to identify an impurity.

Information about the dyestuff can be obtained from its melting point range and the manner in which it melts. Anthraquinones, for example, usually melt at much higher temperatures than azo dyes. Metallic dyes for nylon have very high melting points, if they melt at all, and they usually suffer decomposition.

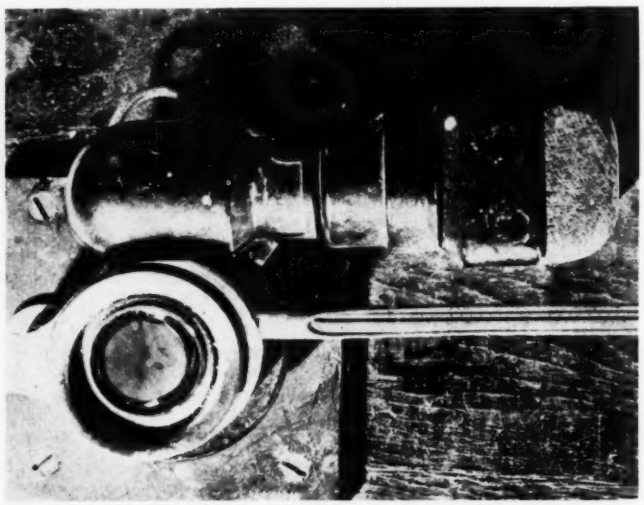
One more physical test that can be performed on the dyestuff even before isolation involves its manner of dyeing on multifiber cloth. Although most acetate dyes have specific fiber affinity for nylon and some for wool and silk, the reverse is not true. Alizarin cyanine green, a disulfonated anthraquinone dye for wool, also dyes nylon but not acetate. The mono azo metallic complexes for nylon have affinity also for wool and silk but leave acetate pure white. Proper evaluation of the dyeing of multifiber cloth may show a commercial dye to be a mixture of two or more dyestuff components and thus reveal possible structures of its unknown components.

CHEMICAL ANALYSIS

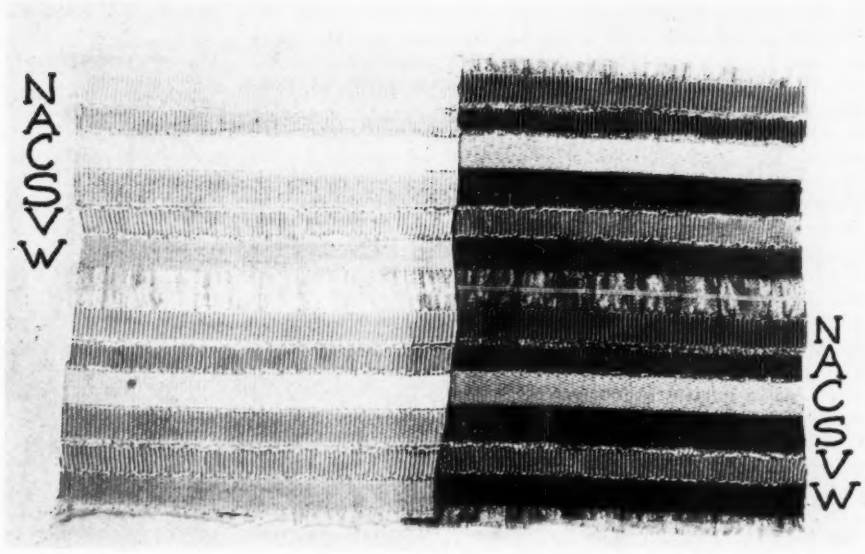
The most fundamental chemical analysis that must be run on the pure extracted dye is that for the elements present. These may include carbon, hydrogen, nitrogen, sulfur, and halogens (including fluorine), as well as metallic ions of the transition series, which may be identified by qualitative tests. The metals most commonly found are cobalt, iron, nickel, copper, and chromium. If a metallic dye is suspected from physical examination, its metallic ion can readily be identified by spectrographic analysis. A small sample of the dye is placed in a carbon arc or flame, and its spectrum observed. Identification of such spectra is a science in itself. Spectrographic analysis of dyestuffs deserves more extensive use (*20*). Examples of this method will be discussed later in this paper.

Qualitative tests for the elements are carried out by any one of the fusion methods employed in qualitative organic analysis. Fusion with sodium or with alkaline carbonates (magnesium or potassium) are the two procedures most widely employed (*27*). The filtrates from the fused mass may then be tested for sulfur, nitrogen and the halogens. For sulfur, the lead acetate or sodium nitroprusside tests are adequate. Any one of the ferric chloride tests should reveal the presence of nitrogen, but it is always wise to run at least two or more of these tests (*27*). Tests should be made for the individual halogens if the Beilstein or silver nitrate tests have been positive, although only chlorine and fluorine usually find application among dyes for man-made fibers.

If a metallized dye is suspected, either the metal must first be split out before quantitative analyses are made, or its presence must be taken into account by weighing the ash from the carbon-hydrogen combustion chamber. Since tests other than analysis for the elements might be desired on the metallized dye, the dye should be separated from its metal. This can be done by taking a sample of the dye and heating it in 10 to 20 per cent hydrochloric acid. This treatment usually splits the metal from



Looking through magnifier of melting point block at crystals on heated block.



Multifiber cloth, undyed and dyed, showing differential dye pickup on Nylon, Acetate, Cotton, Silk, Viscose and Wool.

the complex and leaves the unmetallized dye intact for further chemical tests.

The amounts of carbon and hydrogen in a sample are always measured simultaneously by microanalysis. A weighted portion of the sample is burned in a combustion chamber, the carbon of the sample being measured as CO_2 absorbed, and the hydrogen in terms of the water formed and absorbed. For most cases the method is simple, fast, and accurate.

The entrance of fluorine into the dyestuff field has been heralded by the appearance in the last few years of many patents covering the structure of fluorine-containing dyes. Fluorinated intermediates have raised the need for more precise dyestuff analyses. The filtrate from the fusion process can be tested qualitatively for fluorine with calcium chloride; in a positive test, gelatinous calcium fluoride should precipitate. The silver nitrate method, applicable to the other halogens, cannot be used, since silver fluoride has high solubility in water. Another qualitative test for fluorine is one which is very simple and highly effective. A small sample of the unknown compound is dissolved in concentrated sulfuric acid in a small, *clean* test tube. The contents of the test tube are then heated in a Bunsen flame to a boil. If the original sample contained fluorine, the sulfuric acid solution will fail to wet the sides of the test tube. This effect is caused by the action of the hydrogen fluoride which is split out. The most effective and positive qualitative test for fluorine is based on the spectrographic analysis of a small sample of the unknown dye ground in a mortar with calcium sulfate (*9*). If the original sample contained fluorine, the calcium fluoride band will appear.

Although the qualitative test for fluorine in a dye is a simple one, few laboratories in this country at present are equipped to do accurate quantitative fluorine determinations. The problem is a complicated one. The presence of fluorine in a compound will often affect the accuracy of the carbon and hydrogen analysis because the carbon-fluorine bond is extremely stable. The analytical procedures which have been published all have their specific advantages as well as their individual drawbacks. If only one fluorine atom is present in a structure, the classic lead chlorofluoride determination is adequate (*26*). When two or three atoms of fluorine are present in the structure, other methods are more suitable. The sample can be decomposed in a nickel bomb and the distilled fluoride may then be titrated with thorium nitrate (*14*).

In the past year, H S Clark (*5*) published an excellent microanalytical method for the determination of fluorine in organic compounds. This method involves combustion of the organic substance at 900°C with oxygen in a fused quartz tube, followed by an acid-base titration using phenolphthalein as the indicator. A second titration, after the addition of mannitol, is necessary to account for the fluorine of a boron-fluorine combination. Although other halogens and sulfur do not interfere, nitrogen does, and thus the use of the method is limited. An excellent summary and bibliography of the various decomposition methods which have been attempted have been given by Elving and Ligett (*7*).

The quantitative procedures for the analysis of chlorine and sulfur are usually extensions of qualitative procedures. The sample is usually decomposed in a Parr bomb and the halogens are then measured as their silver salts and sulfur as barium sulfate. Some organic compounds containing sulfur or chlorine can be more advantageously analysed by catalytic combustion. The decomposition is carried out at high temperatures in a stream of oxygen, the sulfur being oxidized to sulfate which can then be precipitated as barium sulfate. The chlorine is measured as its silver salt. Volumetric procedures for these elements, after decomposition has been effected in the furnace, work equally well.

The Dumas method for nitrogen is perhaps the most widely used microanalytical procedure for the quantitative analysis of this element. This principal limitations of this method lie in its use for determining nitrogen in heterocyclic compounds, certain isocyanates, and other specific examples found in the literature (*13, 17, 28*).

DETERMINING DYE STRUCTURE

Once the analysis of the dye is complete, it must be classified into a definite group such as azo, anthraquinone, diphenylamine, etc. Since this article is restricted mainly to acetate and nylon dyes, only the structures suitable for these dyes will be discussed.

Perhaps the reactions which are most suitable for dyestuff classification and about which most has been written are those which subject the dyestuff in question to the action of various reducing agents before subsequent reoxidation. The table prepared by A G Green (*12*) will be given below, principally to show the broad application of such a scheme for the classification of dyes of any basic structure. A similar system was used by Rota (*21*) who, however, used stannous chloride and

hydrochloric acid for the reduction of the dyestuff, and ferric chloride as the oxidizing agent. Although many of the basic dyestuff structures listed in Table II will dye acetate and nylon, they are not considered acetate dyes for they lack the required fastness properties.

The reduction scheme most suitable for the classification of these dyes is one which employs different reduction procedures. Which reduction procedure should first be done depends on what the investigator has already found out about the structure of the dye. The stannous chloride-hydrochloric acid technique is excellent for azo dyes because this reagent mixture will reduce the azo groups to two amines which may then be separated and identified. Separation and crystallization of the amines as hydrochlorides can often be accomplished by addition of alcohol to the strongly acidic mixture. The hydrochlorides can then be separated from each other by fractional recrystallization. If an azo dye is suspected because of a loss in color on reduction, which cannot be restored by oxidation, immediate positive test for the azo group should be made. This simple test is based on the fact that azo dyes, when dissolved in concentrated hydrochloric acid, give color changes on the addition of sodium nitrite, due to the formation of an azoxy compound.

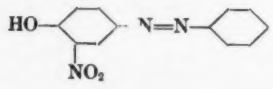
The dye being investigated should be subjected to the vat test. This requires reduction with hydrosulfite in caustic and reoxidation, either by neutralizing and blowing air into the mixture, or by adding persulfate. When the hydrosulfite reaction is employed, strong acidification should be avoided, since free sulfur will precipitate and contaminate the sample, which could otherwise be used to carry out other tests. This reduction is preferred for anthraquinones, for it avoids the danger that the reduction may be carried beyond the *leuco* state to the anthrone or anthacene, as is possible with less specific or stronger reducing agents.

Another reduction reaction which should be employed on the unknown is treatment with sodium sulfide in ammonium hydroxide solution. This reaction is very specific for nitro and nitroso groups present in a dye molecule. Since all diphenylamine and many azo dyes have nitro groups, this reduction is imperative. If the nitro group is present on the nucleus of an azo dye, the resulting product of reduction will still have color, although it may differ from that of the original, since its principal chromophore, the azo group, remains intact. Analysis of the reduction product will now tell the investigator not only the molecular weight of the original dye, but exactly how many nitro groups were present on the original dyestuff nucleus. Since the product of reduction of a nitro group is a primary amino group, the investigator has an additional valuable and versatile key to work with. The amino group can be acetylated, methylated, or even diazotized and coupled further into another known coupling component. All the resulting structures can be analyzed if desired but these procedures are usually not necessary at this point of the analysis.

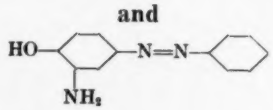
If the nitro group is present on the nucleus of a diphenylamine dye, the resulting product of reduction should be colorless, since all chromophoric value has been lost and the resulting primary amine, itself only an auxochrome, cannot give color to the molecule (*25*). An analysis of the reduced product and a comparison of the ntirogen ratio to the total molecular weight will serve to identify the original dye as a diphenylamine. Postive classification can be accomplished by making derivatives of the dye through reaction with the available hydrogen of the secondary amine. The Schotten-Baumann reaction, giving an acetyl derivative which can be recrystallized and analysed, should be adequate.

If the unknown has been classified as an azo dye, its color in concentrated sulfuric acid and its color on drowning in water may tell something about, or even identify, the structure if the latter has been recorded by previous investigators. F M Rowe, editor of the *Colour Index*, should receive particular mention for his work on the use of concentrated sulfuric as a means of identifying azo dyestuff structures (*22, 23*). The greatest limitation of this system is that many more dyes have been synthesized and marketed since 1920, when the literature was most recently summarized. The color of these newer dyes in sulfuric acid is not known; nor is that of the new structures that are being patented every year. A thorough study of the work published on the colors of the various dyes in sulfuric acid will, however, give the investigator a very generalized and relative means of knowing something of the dyestuff structure (*4*).

The greatest problem faced by the investigator in dealing with an azo dye is splitting the azo group and satisfactorily isolating and identifying the products of reduction. The following examples

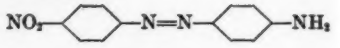


and



would be simple since the products of reduction in both cases are identical: aniline, a liquid, and 4-hydroxy-*meta*-phenylenediamine, a solid. No separation problem would be present. If the proper reduction test were followed, the two could be differentiated and their structures would be known.

The compounds



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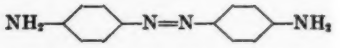
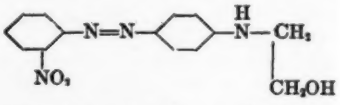
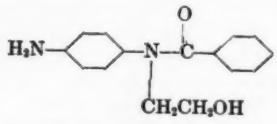


would both give one and the same product—*para*phenylene diamine.

Progressing to more complex structures,

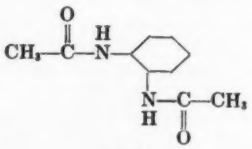


would give *ortho*-phenylenediamine and *para*-amino phenyl ethanolamine. Two products like these might prove difficult to separate. Two distinct procedures could be tried. The secondary amino group could first be treated with benzoyl chloride and then the azo group reduced catalytically. The resulting products, *ortho*-phenylenediamine and



could more easily be separated.

Catalytical reduction could be carried out in an acetic anhydride medium affording



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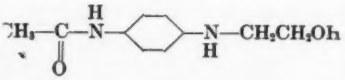


These compounds might be separated more easily than the free unsubstituted amines.

For azo dyes which cannot be readily reduced, a very useful method is that of distillation with soda lime. The color is ground with four times its weight of soda lime and the mixture is distilled. A fission of azo group occurs and any amides present are hydrolyzed.

$$RN{=}NR' \rightarrow RNH_2$$
$$RNH{-}COR' \rightarrow RNH_2$$

The amine so obtained distills over, unless it contains a nitro group or hydroxy group, also present in the nucleus (*6*).

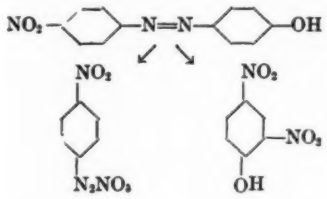
Knecht (*15, 16*) reported a method of estimating azo dyestuffs with titanous chloride. This method consists in titrating the

azo dyestuff with a solution of titanous chloride:

$$TiCl_3 + HCl \rightarrow TiCl_4 + H$$

It follows that each azo group (-N:N-) requires four moles of $TiCl_3$ for complete reduction. This method has very limited application since the majority of the acetate azo dyes are mono azos.

Sometimes the splitting of the azo group is not achieved satisfactorily by reduction, particularly if there are large groups *ortho* to the azo link. Oxidation with fuming nitric acid may then serve as an excellent method (*24*). This oxidation yields the diazonium nitrate of the amine base originally used in the coupling and the nitrated derivative of the coupling component.



The reaction is a general one. Azo compounds containing NH_2, OH, OCH_3, NO_2, etc., groups in a position *para* to the azo group are decomposed smoothly by this means. These substituents in the *ortho* position limit the reaction, unless there is another group in the *para* position. In the case of *meta* substituents, the reaction is very limited. Azoxy compounds containing an amino group *para* to the azoxy readily yield the diazo compound.

The importance of this reaction as a tool in azo analysis cannot be overestimated. The unknown dye can be split and the base coupled into a known coupling component such as *ortho*-cresol. The resulting dye is then purified and analysed. Since one-half of the dye (the *ortho*-cresol) is known, the other half can be deduced by difference from the total analysis. The base postulated by this difference is now also coupled into *ortho*-cresol and the resulting product after purification should check the unknown both in mixed melting points and analysis, if necessary.

This reaction can further tell the investigator whether a particular element found to be present in the structure of the original unknown dye is in the amine base portion of the molecule or in the coupling component. If the new dye made by coupling the diazonium nitrate of the original amine base into *ortho*-cresol now contains the element in question, the investigator knows that the element X must have been present originally in the amine base side of the azo dye molecule. If not, the remaining nitrated derivative may be analyzed for the element in question.

Rowe and Levin have made a study of azo dyes formed by coupling diazotized

TABLE II

SCHEME FOR CLASSIFICATION OF DYES BY OXIDATION-REDUCTION REACTIONS

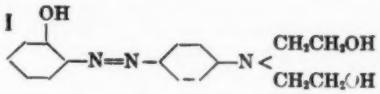
Decolorized by zinc dust			*Unaltered by zinc dust or only slowly or partially reduced*	*Changed in shade but not decolorized by zinc dust*
Color restored on exposure to air	*Color restored by oxidizing agents*	*Color not restored by oxid'n*		
Azines	Triphenylmethane dyes	Nitro,	Quinolines	Anthraquinones
Oxazines	Pyrones (red)	Nitroso and	Thiazoles	
Triazines		Azo dyes		
Indigoids				
Pyrones (violet and blue)				
Acridines				

bases with β-naphthol and with Naphthol AS. Their findings have developed into a means of azo dye identification (*23*). Included in their scheme is the coupling of the diazonium nitrate obtained from the oxidation with fuming nitric of an unknown azo dye and coupling it into β-naphthol and Napthol AS. The greatest drawback of this procedure is that their work has not been extended to include the modern azo dyes. The choice, β-naphthol and Naphthol AS, could be improved upon, since these coupling components often afford highly insoluble products which are difficult to purify by recrystallization and have very high melting points. The cresols are probably more satisfactory for this work.

Palkin and Wales (*19*) have described a similar method for the identification of many of the phenols used as coupling components. Diazotized *para*-nitraniline was the reactant. This work too, as well as that of Forster and Hanson (*9*), has not been brought up to date.

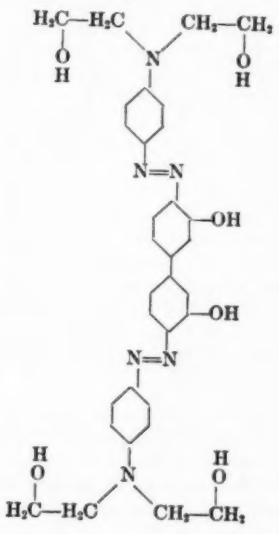
It might be well at this point to caution the investigator against adopting too simple an empirical formula for the dyestuff with which he is concerned. A dyestuff might, from the evidence of elementary analysis, have a structure such as:

I

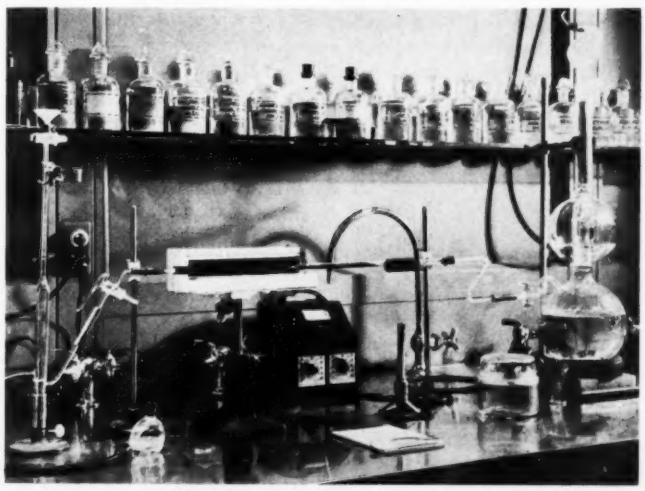


but may actually turn out to be

II



This type of error could only be made on



The Dumas apparatus is often used for quantitative determinations of nitrogen in organic dyestuff molecules.

the basis of an incomplete investigation. It would also be more likely to appear in the investigation of azo pigment structures, since acetate affinity, or lack of it, would tend to eliminate a great many structures of type II.

When investigating acetate dyes having anthraquinone basic structures, we are fortunate in knowing that it is only those substituted at the *alpha* positions which have affinity for acetate fiber. We further can assume that the substituents we expect to find at the 1, 4, 5 and 8 positions are amines (primary, secondary, and sometimes, tertiary), phenols and substituted phenols, such as anisols, and sometimes, halogen atoms. When functional groups are present on the anthraquinone nucleus, at positions other than *alpha*, they influence the shade and other properties of the dye in question. Mono-substituted amino anthraquinones are usually red in shade, and the diamino substituents are violet in shade, while the tetra-amino substituents fall farther over into the blue side of the spectrum. Mono-hydroxy substituted anthraquinones are yellows with very poor color value. Dihydroxy substituted anthraquinones are red and orange. An anthraquinone dye containing a mixture of an amine and a hydroxy group would therefore be predicted to fall in between. This prediction is borne out in practice, as demonstated by the many such dyes red-violet or pink in shade.

In order to determine what substituents are on the principal dyestuff nucleus, certain basic reactions should be carried out. After the investigator has determined the type of dye structure he is dealing with, the compound should be both acetylated and methylated, recrystallized and reanalyzed. This tells the investigator exactly how many primary and secondary amino groups or hydroxyl groups are present in the molecule. The methylation should be carried out under vigorous conditions so as to replace all the available hydrogens by methyl groups. Diphenylamine is a secondary amine and can be methylated.

Should an azo dye lend itself to methylation, and the methylated group found when the azo link is spit, the investigator knows on what group, and on what side of the molecule, the methyl group had attached itself. When there is a question whether the methylation has affected an amino or hydroxyl group, the methylimide determination should be made. This highly specific analysis shows that the methyl group has attached itself to a nitrogen and not to an oxygen atom.

Now that physical and chemical analyses have pointed toward a definite structure for the unknown dyestuff, a literature and patent search is in order. This search should be made after the analytical data are complete so as to avoid taking the wrong trail on earlier inconclusive evidence. The right trail is difficult enough to follow because many patents never give a specific example of the dyestuff suspected, but include its structure in a very general category.

In the case of a very new dyestuff, the patent application files may be the only public source of information. If the structure has not been patented, or is not patentable, it may still appear in a patent as a process example, where other structures appear as product claims. Should the investigator be successful and find the structure he suspects, he will then have found a method of synthesis which he may try. Ultimate synthesis is the only sure proof of structure. The synthesized dye can be checked with the unknown, by mixed melting points, and mixed melting points of its derivatives, by comparison of spectrophotometric curves, and even, if desired, by dyeing from dispersions of equal strength.

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OPENING FOR MATERIALS ENGINEER

The Bureau of Aeronautics, Department of the Navy, at present has an opening for a Materials Engineer (Textiles) at a salary which may range, depending upon the experience of the individual, from $5060 to $7040 a year. The position reportedly offers a wide variety of experience and extensive contacts in the industry. Location: Washington, D C.

This engineer will be responsible for the development of flight and special protective clothing for naval aviation personnel as well as the development of cordage, ropes, webbing, duck, and other fabrics used in naval aircraft and aircraft equipment. He will also act as technical advisor to other sections of the Bureau and field activities on problems involving textile materials. His work will involve close and frequent contacts with textile and clothing manufacturers and the Defense establishments.

The position requires a general education in textiles and a practical familiarity with a wide variety of fabrics. Experience in the design and fabrication of clothing is highly desirable but not essential.

Interested parties should contact Gene T Arnould, Head, Employment & Employee Relations Units, Dept of the Navy, Bureau of Aeronautics, Washington 25, D C.

Proceedings of the
American Association of Textile Chemists and Colorists

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Editor of Proceedings: Carl Z Draves, 30 Lahey St, New Hyde Park, N Y

September 15, 1952

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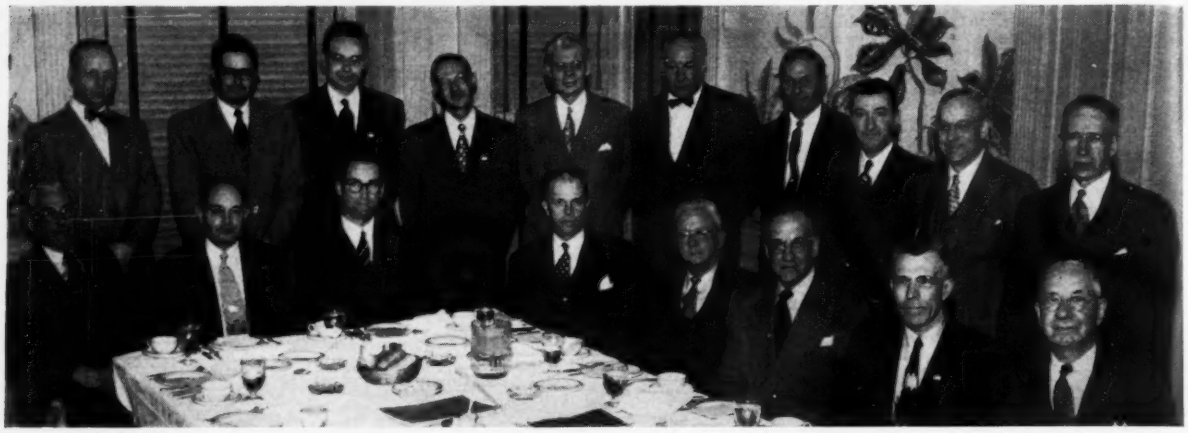

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52-19

Education: A B, organic chemistry, followed by textile school.

Experience: Two years as chemist in product development.

Age: 29; married; references; vicinity of Philadelphia preferred.

9-1, 9-15

CALENDAR

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(See Council dates)

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MID-WEST SECTION

October 4, February 14 (Hotel Bismarck, Chicago, Ill); April 18 (Netherland Plaza, Cincinnati, O); June 20 (Outing, Lake Delavan, Wis); November 7, 1952 (Hotel Bismarck).

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September 26, October 24, November 21 (Kohler's Swiss Chalet, Rochelle Park, N J); January 30, February 27 (Hotel Statler, New York, N Y); April 17, May 22 (Swiss Chalet)

NORTHERN NEW ENGLAND SECTION

December 6 (Annual Meeting, Lowell Textile Institute).

PACIFIC SOUTHWEST SECTION

October 4 (Outing, Ojai, California).

PHILADELPHIA SECTION

Oct 24 (Penn Sheraton Hotel); Dec 5, Jan 16 (Kugler's Restaurant).

RHODE ISLAND SECTION

Oct 24 (Providence Engineering Society); December 5 (Annual Meeting, Sheraton-Biltmore, Providence, R I).

SOUTH CENTRAL SECTION

December 6 (Patten Hotel).

WESTERN NEW ENGLAND SECTION

Oct 3, Nov 14 and Dec 12 (Rapp's Restaurant, Shelton, Conn).

OTHER EVENTS

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1953 Annual Meeting, June 29-July 3, Chalfonte-Haddon Hall, Atlantic City, N J.

AMERICAN STANDARDS ASSOCIATION

34th Annual Meeting, November 19, Waldorf-Astoria, New York, N Y.

INTERNATIONAL COTTON AND RAYON EXHIBITION

Sept 27-Oct 12, Busto Arsizio, Italy.

NEW YORK BOARD OF TRADE (DCAT)

26th Annual Meeting: September 25-28, Pocono Manor Inn, Pocono Manor, Pa.

27th Annual Drug, Chemical and Allied Trades Dinner, March 5 (1953), Waldorf-Astoria, New York, N Y.

TEXTILE RESEARCH INSTITUTE

23rd Annual Meeting, Nov. 17-18, Hotel Commodore, New York, N. Y.

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HYDROGEN PEROXIDE BLEACHING OF TEXTILE MATERIALS*

A Background of Present-Day Procedures

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INTRODUCTION

THE background of the developments in hydrogen-peroxide bleaching, particularly of those in which there may have been some unwitting contributors, is not just a chronological tale of progress. For over a quarter of a century this writer has been fortunate enough to play an active role in the advance of the techniques of hydrogen-peroxide bleaching. It now seems appropriate to review some of these advances and to reveal certain hitherto unpublished accounts of how they came about.

HISTORICAL —— In a paper over twenty-five years ago, this writer pointed out five variables that should be taken into account in any kind of peroxide bleaching: hydrogen-peroxide concentration, total alkalinity, pH, time, and temperature. At that time peroxide bleaching was a somewhat confused and hazardous art, and the results obtained were fairly hit and miss. Since then the following additional variables have had to be taken into account: the previous history of the textile material, that is, the kind of treatment it has already received before coming to the bleachery, the suitability of the existing equipment, the quality and available quantity of process water, and general local conditions, which include the end use of the bleached material, the quality required, the allowable time for the bleaching operations, the production requirements and, last but not least, the cost.

All of this sounds forbidding and complicated, but, fortunately, there are available experience, skill and a willingness to co-operate with the user of hydrogen peroxide in co-ordinating all of these variables to produce a satisfactory result in a large majority of cases.

Hydrogen-peroxide bleaching, today, is being done more or less extensively on the following textile materials: cotton raw stock, skein and package yarns, knit goods and piece goods; wool raw stock, felt, yarns, piece goods, knit goods and felt hats; worsted tops, piece and knit goods; silk piece and knit goods; jute stock, yarn and fabrics (burleys); linen yarns and fabrics; ramie, hemp, grasses, straws, and other miscellaneous fibrous materials; rayons, acetates, and other synthetic or man-made fibers, alone or blended with natural fibers.

H G Smolens

An American pioneer in peroxide bleaching, who has participated in the development of modern continuous and batch peroxide bleaching methods, compares present-day procedures for wool, cotton, and other fibers with bleaching in the twenties and thirties. He names the variables that must be considered in a bleach bath: namely, peroxide concentration, total alkalinity, pH, time, and temperature. Certain other factors not concerned with the bath itself also require attention, such as history of the textile, nature of the equipment, water quality, other local conditions, production requirements, and cost. The principal emphasis in this review has been placed on the historical development of the cold peroxide bleach for cotton and wool, steam-bleaching cotton, and the white-scour process for wool.

It should be obviously that, with such a variety of textile materials and variables, there is not, indeed there cannot be, a set formula or series of formulas, or a routine procedure for bleaching with hydrogen peroxide. This means that, for the best and most efficient use of hydrogen peroxide in any particular bleachery, on any particular textile material, it is advisable to get the best technique, or, if you please, a "custom service", rather than formula by mail or a procedure copied from some other bleachery. Such a service is available to all who wish it.

BLEACHING CHEMICALS —— In general use today are the following peroxide-bleaching primary chemicals: 27.5% hydrogen peroxide (100-vol), 35% hydrogen peroxide (130-vol), 50% hydrogen peroxide (197-vol), several silicates of soda, phosphates, pyrophosphates, polyphosphates, caustic soda, soda ash, and ammonia water. There are available complete and simple methods for testing the bleaching value of these chemicals, so that peroxide bleaching can be done in a fairly scientific manner, even by bleachery operators of ordinary skill. There are also available equipment and methods for determining the quality of peroxide-bleached textile materials in comparison with established standards. All of this has made peroxide bleaching no more difficult or complex than other wet-processing operations, such as dyeing or finishing.

You will note the listing of soda ash as a primary peroxide-bleaching chemical. It was in 1929 that this writer discovered that a softer knitting yarn could be produced in package-machine bleaching with hydrogen peroxide by replacing silicate/caustic alkalinity with silicate/soda-ash alkalinity. There was quite a little objection to this among some bleaching technicians, and at least one paper, complete with charts and curves, was published warning against such a bleaching formulation and showing that the peroxide stability was lowered to such an extent that no satisfactory result could be obtained. However, there is today very much more soda ash than caustic soda used in batch-bleaching cotton stock, yarns, knit goods, etc, because the resulting quality of the bleached material is more satisfactory for most purposes.

* Presented before the Midwest Section in Cincinnati, Ohio, on the afternoon of April 19, 1952.

In the notes that follow the bleaching of different fibers is discussed.

COTTON

In the early 1920's, some cotton yarn in skeins, after a thorough scouring, was being bleached by an overnight submersion in peroxide solutions copied from wool bleaching. Efforts were being made to peroxide-bleach package yarn in the new package dyeing machines, originally made of iron, later of Monel metal. The packages were first scoured, then rinsed, and finally peroxide-bleached. The process took a lot of time and in many instances did not produce satisfactory work. Peroxide bleaching was also being tried out in kiers on cotton piece goods, mainly colored-yarn decorative fabrics, such as towelling and shirtings.

In spite of many discouragements, the work was carried on, and soon it was found that cotton could be scoured and bleached simultaneously with a hydrogen-peroxide bleaching solution containing caustic soda in limited amounts at temperatures around 180° F. This type of procedure found immediate acceptance in package-yarn bleaching, especially for knitting yarns, for the result was excellent and the over-all cost reasonable. It was not so quickly accepted by fabric bleachers for a variety of reasons, such as lack of absorbency of the bleached goods, trouble with rust and other stains from iron kiers, pumps and piping, the use of steam-injector kiers, and higher chemical costs. However, the hydrogen-peroxide kier-bleaching process began to make considerable headway on colored-yarn fabrics, even though some of the vat colors used in yarn or stock dyeing were not very fast to peroxide bleaching. Also, some of the colors supposedly vat-dyed were matched by topping with direct colors, which are a bane to any bleachery. The process also became attractive for very light-weight cotton goods when it was found that they could be caustic-boiled, washed and then hydrogen-peroxide-bleached in the same kier, thereby stopping a great deal of pulling and fraying that took place when such goods had to be pulled repeatedly through tight washers at high speeds. By the end of the 1930's a large percentage of colored-yarn fabrics and a considerable percentage of lighter-weight white fabrics were being peroxide-kier-bleached with excellent results in every respect and at reasonable costs.

Early in the 1920's there was very little peroxide bleaching of heavy cotton goods that had to be kept in open width, but some jig work was being done in bleacheries that had experience on silk-cotton fabrics, which were also handled in open width.

In the spring of 1928 this writer was asked to do something about preparing heavy goods like drills and ducks for mineral dyeing. The existing procedure was to pad the singed goods with a hot caustic solution, fold them down in boxes overnight, wash in the morning and dry for dyeing. This was satisfactory for dark shades, but orders had come in for light tans and greys. These shades not only looked muddy but did not cover the motes which showed up as black specks on the dyed goods. In our first experiments the goods received the usual caustic steep and wash and were then repadded with a hydrogen-peroxide solution at room temperature, steeped for several hours more and again washed. The result was certainly amazing in that the goods were brighter and the motes much lighter and softer. All light-color mineral dyeings on the goods prepared in this manner were excellent. Good results were also obtained in the direct, vat and sulfur dyeing of heavy fabrics prepared in the same way. Several months later, the two steepings were combined in one operation, and the singed goods were padded at room temperature in a single bleach bath containing a strongly alkaline hydrogen-peroxide solution, steeped overnight and then washed for dyeing. This process, called the "Cold Peroxide Bleach Process" because it was done at room temperature, has been of tremendous help to almost every bleachery in this country during the past twenty years and is still being used very extensively on a wide variety of fabrics. Although the chemical cost is relatively high, the saving in other directions more than compensates for this.

In the spring of 1930 a continuous kier was built and set up for a test run in a large southern bleachery. This consisted of four Monel-metal J-boxes in series, all resting in a single large steel tank, for caustic scouring, a steam recuperator for washing, and two additional Monel J-boxes in series, resting in a smaller Monel tank, for peroxide bleaching. The J-boxes were perforated below the solution level to allow freedom of flow. Pumps sprayed boiling caustic over the top of the caustic J-boxes and on to the goods folded in open width, moving through them continuously at about sixty yards per minute. Other pumps sprayed a warm peroxide bleaching solution over the top of the peroxide J-boxes onto the goods passing through them. Although the results were fairly good, the chemical and operating costs were high, and the project was abandoned. This writer, who was present to watch the peroxide-bleaching end of the range, noticed that the goods, even though well scoured and bleached, still carried out of the treating tanks the full strength of the treating solutions, which were then immediately washed down the drain and wasted. One morning, while the continuous kier was not in operation, he took some of the caustic scoured goods, just dipped them in the peroxide solution to wet them out (instead of bleaching them in this solution as usual), squeezed them to remove excess solution, and then held them in the path of a stream of high-pressure steam. The resulting bleach was both extraordinary and instructive. Many additional samples were made in the same manner and during the succeeding months were called to the attention of quite a few bleachery technicians and executives, but no one was interested. Additional discouragement came when a leading bleachery-machinery firm estimated a price of $90,000 for an experimental machine for steaming goods, and when steam experts estimated a cost of from $25 to $50 per ton just for heating bleach-saturated goods. However, experimental work was continued, mostly on heavy goods in open width. Finally, in 1937, at a finishing plant in Philadelphia, the first stainless-steel J-box for open-width bleaching was built and operated, thus really proving that this process, called "Steam Bleaching", had considerable merit.

From this beginning, unpublicized and certainly not resulting from any definite encouragement, has come the present continuous bleaching process, which has just about revolutionized cotton-goods bleaching, not only in this country but throughout the entire textile finishing world.

Today, especially in the western hemisphere, a very large percentage of all cotton goods bleached for white or for dyeing, and of colored-yarn decorative fabrics, is going through many continuous bleaching ranges at speeds up to 300 yards per minute. The speed limit is not in the bleaching but in the washing. There are rope and open-width continuous-bleaching J-boxes in operation and also conveyor-type ranges for heavy goods that have to be handled open width without wrinkles. Excellent work is being done on all classes of goods.

It is this writer's opinion that steam bleaching, in spite of the many improvements made in the past five or six years, is still subject to major improvements.

Tubular knit goods and warps can be bleached with hydrogen peroxide to an excellent whiteness with low weight loss, fairly low wash shrinkage, and small loss of natural softness with the result that little or no chemical softening is required. The best work today is done in

pump-circulating kiers, but they should be specially designed to allow thorough washing of the bleached goods right in the kier. The steam bleaching of knit goods has not been so successful as that of piece goods, but this condition may be changed in the next few years as the result of newly designed equipment and procedures.

There are quite a few processes in use now for peroxide-bleaching cotton textiles, in which a combination of several procedures is most suitable for special local conditions. The most important of these is the combination of the cold peroxide-bleach process with either a hot peroxide bleach or a steam bleach.

WOOL AND WORSTED

In the middle 1920's peroxide bleaching of wool and worsted textile materials was done mainly by an overnight submersion of the scoured material in a hydrogen-peroxide solution in wood or lead-lined tanks. To keep the materials completely submerged, boards, ladders, ropes and even stone slabs were used. In the morning, after rinsing, the peroxide-bleached material was smoked, where at all possible, with sulfur dioxide gas produced from burning sulfur. What a job it was to raise a large bag full of dripping-wet bleached wool out of a standing bath, and what a Herculean task it was to hold fabrics like heavy wool coatings under the surface of the bleach bath while they were trying their best to push up and out of the solution. This writer has seen strong wood ladders broken in two by heads of worsted yarn that just would not stay down all night, even though several men had worked hard the previous evening to get them fully under the surface. It was certainly a distasteful experience to have to go into a sulfur house, supposedly blown free of sulfur dioxide, and to pull worsted piece goods from glass pins on which they had been hung to keep them from staining from contact with wood or metal. Happily, there has been much progress, and this kind of hard and disagreeable work is unnecessary today.

Wool and worsted piece goods are now bleached in dye kettles or similar machines, or they can be cold-peroxide-bleached by a procedure similar to the one outlined for cotton goods. Worsted top on spools or in balls is bleached best by the cold peroxide-bleach process. Wool and worsted skein yarn is bleached in a Hussong-type machine, in a Klauder Weldon machine or by the continuous-scour and cold-bleach process that has been successfully employed by a large worsted-yarn processor for nearly 20 years. Wool and worsted knit goods are bleached in string-dyeing machines and wool hosiery, in paddle or rotary dyeing machines. Wool and worsted materials that have been chlorinated for shrink resistance can be hydrogen-peroxide bleached very well, but some changes in the ordinary wool-bleaching procedure may be necessary for best results. Wool stock is being bleached in Hussong-type machines, in stock-dyeing machines with pump circulation and in wool-scouring ranges, in which the scoured wool passes through a peroxide solution in the last bowl just before going into the dryer. In this latter process, the whitening of the wool continues for at least 24 hours after the wool has been dried, and there may be traces of peroxide in such wool for days or even weeks after the bleaching. Sometimes this trace of peroxide can cause trouble in the dyeing of the bleached wool, but this difficulty is easily overcome.

The "White Scour Process", as this writer likes to call the continuous scour and bleach had a very curious beginning. In the middle 1920's a prominent wool-scouring firm had an English chemist who showed them a way of putting a luster or bloom on scoured clothing wools by using alum instead of sulfuric acid in the last or neutralizing bowl of the scouring range. The wool really had a beautiful appearance, but when it was made into yarn and cloth that had to be soap scoured and fulled, there was a great deal of trouble in the dyeing, as you can well imagine, from the alum and aluminum hydrate left in the wool by the scourer. This chemist, considerably alarmed by the complaints that poured in, tried adding various chemicals, including concentrated hydrogen peroxide, with the alum. Nothing did any good in the way of improving the scourability of the wool, but he noticed that the addition of hydrogen peroxide whitened the wool appreciably. So, he did the logical thing—he omitted the alum but kept the peroxide. It was some years before the other wool scourers got on to this trick, but they finally did.

About ten years later this writer was called in to a large carpet mill to advise on the whitening of stained locks in South American carpet wools, which were being hand-picked. The white scour process was suggested and samples were made. All of these were quite satisfactory, but the mill management would not make the considerable investment in stainless-steel or lead last bowls to their several scouring ranges. Less than a year later, another, more progressive carpet mill tried this white scour on their wool blends and adopted the process with great success. Most of the other wool scourers, mill and commission alike, are using this process in a large way today.

SILK

There is only a very small volume of real-silk bleaching being done today, and it does not look as though silk, as a textile material, will return to its important position of the 1920's. The major improvement in hydrogen-peroxide-bleaching this fiber was in silk and cotton combinations. By keeping the gum on the silk until the cotton was almost fully bleached, not only was considerable chafing and fraying of the silk avoided, but also its tendency to turn yellowish from prolonged treatment in the hot peroxide solution.

JUTE AND LINEN

Jute and linen textile materials are bleached in general with hydrogen-peroxide bleaching procedures similar to those used for specialty cotton bleaching, in which a combination of several processes is required. These invariably take special study and consideration.

HEMP, GRASSES, STRAWS, RAMIE, ETC

Textile materials made from these fibers are used mainly in making women's millinery, and they offer highly specialized problems in peroxide-bleaching techniques.

RAYONS, ACETATES, VICARA, NYLON, ORLON, DACRON, DYNEL, SARAN, ACRILAN, ETC

Textile materials made from these fibers are the subject of considerable work among bleaching technicians today. Synthetic fibers in general are fairly bright and white when manufactured. Most of the discolorations that we find in the yarns or fabrics made from them may be due to staining or yellowing from some of the treatments that the yarns receive in their preparation for knitting or weaving and from the presetting given fabrics for dimensional stability. The whitening of synthetic textile materials therefore seems more a matter of stain removal than of what we usually consider bleaching.

The best method of peroxide-bleaching rayon fabrics is the cold peroxide-bleach process. For acetates, for which the saponification problem is of considerable importance, the newly available peracetic acid is the best peroxygen chemical to use. With the newer synthetics, we are not too happy over the results, to date, of peroxide-bleaching attempts, although

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Mid-West Section——

SOME OF THE VARIABLES CONNECTED WITH THE DYEING OF ORLON*

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INTRODUCTION

THIS paper deals with the dyeing of Orlon acrylic fiber, Type 41. Originally it was felt that a more general type of paper might be of value. However, a review of the literature indicated that most of the articles that had been previously published tended toward this very thing. It was therefore decided to present as much information as possible connected with the dyeing of polymers, such as Orlon.

A literature survey of this field was made, primarily to find the most promising approach to the dyeing of this fiber. It was noted that there were many different approaches to the problem. These approaches were as follows:

1) Dyeing in the presence of amines, amides and phenol *(1)*.
2) Polymerizing acrylonitrile in the presence of 65% solutions of $ZnCl_2$, $ZnBr_2$, or ZnI_2 *(2)*.
3) Dissolving the polymer in a solvent, such as dimethylformamide *(3)*.
4) Modifying the Orlon polymer with polyvinyl pyridine in order to improve dyeing quality *(4)*.
5) Increasing the dyeing temperature to 250°F (15 lb pressure) *(5)*.
6) Dyeing technique dependent upon the types of dyes being considered *(6)*.
7) Dyeing technique dependent upon the type of polymer, Type A-3, Type 81, and Type 41 *(7)*.

The most promising approach to the dyeing of Orlon was the method developed by Feild. This is the much-talked-about cuprous-ion method. This method has considerable merit and deserves attention. The method is applicable to the dyeing of both Type-41 staple fiber and Type-81 filament with acid and direct colors. The method produces heavy shades with selected acid dyes on Type 41 at 212°F (atmospheric pressure); similar shades are more difficult to produce on Type 81 at 212°F without the assistance of a carrier of the complex-phenol type.

G D Fronmuller

Two important variables connected with the dyeing of Orlon acrylic fiber by the cuprous-ion method are type of acid dye and reducing agent. In general it was found that acid dyes containing monosulfonic acid groups are essential for producing good dyeings, while it is necessary to have a reducing agent of sufficient oxidation-reduction potential to reduce cupric to cuprous ion but not to affect dyestuff.

Early work indicated that the method would require variations with the various types of dyestuffs *(8)*. The variables appeared to be pH, percentage of copper salt, the percentage of reducing agent and the nature of the dyestuff. Many of the variables pointed out at that time have since been proven to be valid, and, as is often the case, other variables have been introduced. These appear to be the nature of the Type-41 polymer, the type of reducing agent, the rate at which the cupric ion is reduced *(9)*.

It becomes apparent that it is essential to resolve some of these variables so that a simple approach can be maintained. The two most important variables appear to be the type of dyestuff and the reducing agent.

EXPERIMENTAL

SELECTION OF DYESTUFF —— Blaker and Laucius *(9)* state that the principal requirement for the dye appears to be at least one acid group in the molecule; more than one acid group in the molecule seems to reduce dye affinity. An examination of this inference indicates that the presence of more than one acid group will impede dye penetration. The verification of this will be noted in a study of various dyestuffs.

In the case of acid dyes it is important to have a good red, yellow, orange and blue so that satisfactory color combinations can be utilized for the production of browns, greens, navies, and blacks.

One-percent dyeings of various colors were made in a package machine in accordance with the following procedure:

Material	Type 41-2/24 yarn
Liquor Ratio	1:8
pH during dyeing	3.2
Procedure	10% $CuSO_4$ on wt of fiber (owf)
	3% reducing agent
	1% acid dye

The yarn, prior to dyeing, was scoured with an acidic detergent for 30 minutes at 210-212°F and at atmospheric pressure. The goods were rinsed, cupric sulfate was added, and the solution was circulated at 170°F for five minutes. Then the reducing agent was added and the solution was circulated for five minutes, again at atmospheric pressure, and the dyestuff was added. The temperature was increased to 210-212°F. Samples of the dye liquor were withdrawn at periodic intervals and tested for rate of exhaustion and the amount of copper ion still in solution.

The dyestuffs studied are included in Table I.

From these dyeings it was found that in the case of dyestuffs containing monosulfonic acid groups, a true shade of high tintorial value was produced, whereas, dyestuffs containing disulfonic acid groups did not dye the fiber.

Table I indicates those dyestuffs containing monoacid groups and diacidic groups. It is to be noted in the case of Fast Red A and Azo Rubine that the molecular structure is similar except that the Azo Rubine contains two sulfonic acid groups. A similar situation prevails

* Presented before the Midwest Section in Cincinnati, O, on April 19th, 1952. Also presented on May 30th, 1952 before the Canadian Association of Textile Chemists and Colourists at Galt, Ontario, and printed in the *Canadian Textile Journal*.

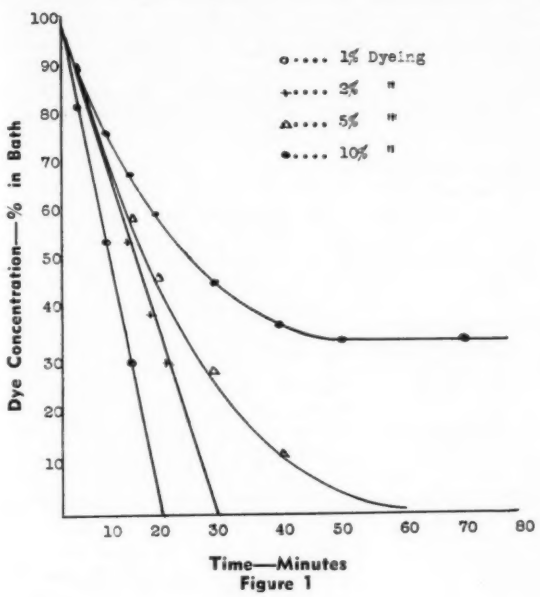

Figure 1

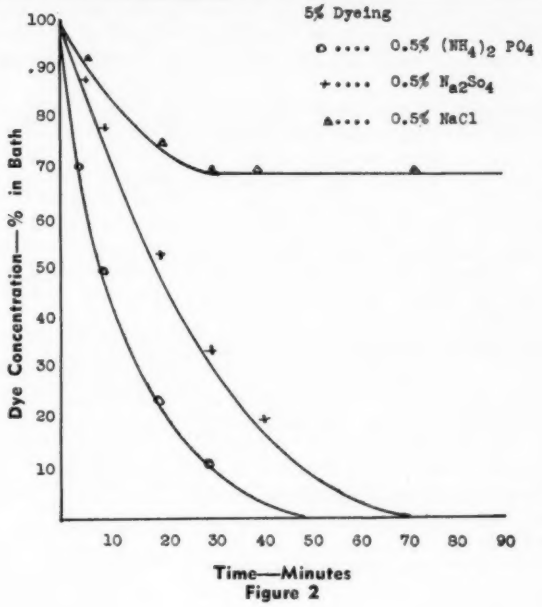

Figure 2

in the case of Brilliant Orange and Scarlet 2R00. These will serve to illustrate the point that in these and similar instances a disulfonic acid group adversely affects the rate at which dye is absorbed.

However, the number of sulfonic acid groups is not the only factor influencing the rate of dyeing; the nature of the substituent groups is also important. To illustrate this point let us consider three yellow dyes having monosulfonic acid groups. Here it will be noted that Quinoline Yellow has the poorest tinctorial power of the three dyestuffs, whereas Metanil Yellow has the highest tinctorial value. If the molecular structure of Fast Silk Yellow GY is compared with Metanil Yellow, it is found that the former contains a nitro group. Tinctorially, these dyes can be used, but when either one is incorporated as part of a mixture, then the rate at which the dye exhausts on the copper complex is important. It was found in the preparation of a navy shade that, in the cases where both Quinoline Yellow and Fast Silk Yellow G were used, the rate of exhaustion of these dyes was greatly impeded.

A continuation of this phase of the study indicated that these yellow dyes, when used in mixtures to produce black, blue and green shades, were retarded as in the case of the navy shade. However, Metanil Yellow exhausted at the rate similar to that of the other dyes used in the mixture.

The mixtures of dyes used to produce the navy, green, black and brown were as follows:

Navy

7.5% Alizarin Direct Blue A Ex Conc
3.0% Orange Y Conc
2.25% Fast Silk Yellow G
2.25% Fast Red A

Brown

6% Xylene Milling Yellow
2% Alizarin Direct Blue A Ex Conc
2% Fast Red A

Green

5% Alizarin Direct Blue A Ex Conc
5% Xylene Milling Yellow

Black

9.5% Alizarin Direct Blue A Ex Conc
4.0% Xylene Milling Yellow
1.5% Fast Red A

RATE OF DYEING———Rate of dyeing was studied with the following dyes:

Fast Red A
Metanil Yellow
Alizarin Blue A E Extra
Orange Y

These dyes were selected for their dyeing properties on Orlon according to the observations above. The dyeing technique previously described was used. The rate of dyeing is shown in Figure 1.

Blake & Laucius *(9)* have discussed the theory of copper dyeing. It was shown by them that the rate of dyeing is affected by the nature of the salts present in the bath. Hydroxylammonium sulfate does not show an adverse effect, whereas hydroxylammonium hydrochloride does. This implies that monovalent anions have an adverse effect, whereas divalent anions do not but are actually beneficial. These authors later contradict this statement and imply that, in the presence of an excess of a divalent sulfate ion, the rate of dyeing is retarded.

Trivalent anions, such as phosphates, were introduced into the dye bath in our experiments. The results are shown in Figure 2.

The indications are that the phosphate ion in these concentrations does increase the dyeing rate 33%, whereas sulfate ion does not reduce the rate of dyeing when

TABLE I

Dyestuff	*Colour Index*	*No of Sulfonic Groups*
Fast Red A	176	1
Azo Rubine	179	2
Orange Y	151	1
Scarlet 2ROO	79	2
Fast Silk Yellow GY	145	1
Quinoline Yellow	801	1 and 2
Xylene Light Yellow	639	2
Metanil Yellow	138	1
Anthraquinone Blue SWF Conc 150% Pdr	Pr 12	?
Neutral Cyanine Green GN	1078	2
Wool Green S	737	2
Brilliant Croceine Scarlet	252	2
Acid Green G	666	2
Fast Green N	...	..
Brilliant Orange	78	1
Acid Yellow	16	1
Cochineal Scarlet	25	1

there is an apparent excess. In the case of the chloride ion there is a very definite retarding effect.

However, in the production of heavy shades an excess of copper ion, reducing agent, and phosphate does not appear to influence the rate of dyeing. The production of black, navy, and green shades required from 10-15% of dyestuff in order to obtain the proper shade. This would seem to indicate that, as fiber dye sites become saturated, it is more difficult to affix dyestuff.

REDUCING AGENT———There are several types of reducing agents available on the reduction of cupric copper to cuprous copper. These may be divided into three divisions: organic, inorganic, and a mixture of the two.

The choice of a reducing agent depends upon the following properties:

1) Solubility
2) Presence of either sulfate or phosphate ions in its composition
3) Ability to reduce cupric ion rapidly
4) Production of sufficient acid to drop the pH of the system from 6 to 2.2-3 upon hydrolysis
5) No adjustment of the pH prior to the reduction of the cupric ion
6) No change in shade from reduction of dyestuff by reducing agent
7) Ability of the reducing agent to produce cuprous copper without producing free or elemental copper

Aside from these requirements there appears to be one other which is related to the oxidation-reduction potential. Oxidation-reduction potentials have been studied, and the indications are that a relatively low potential is required. The method used for measuring oxidation-reduction potentials are most similar to those outlined in Kolthoff's *Treatise on Potentiometric Titrations*. Using the dyeing techniques outlined above and substituting various reducing agents the potentials obtained were as follows:

OXIDATION-REDUCTION POTENTIALS

	millivolts
Zinc sulfoxalate	749.7
Hydroxylammonium sulfate	832.
Experimental reducing agent	497.

Use of the last reducing agent does not materially affect the tintorial power or the shade of the various types of dyes that we used in this study. It was this agent with which most of the dyeings dicsussed above were made.

CONCLUSION

The inferences that can be drawn from this work, which admittedly only covers a relatively small area of the field, are as follows:

The dyeing of Type-41 Orlon acrylic fiber can be accomplished without the use of pressure.

The use of selected dyes of relatively low molecular weight and in which only a monosulfonic acid or carboxylic acid group is present is desirable in dyeing Orlon. The presence of disulfonic acid or dicarboxylic acid appears to impede the rate of dyeing. This effect may be due primarily to the presence of excessive amounts of sulfate ions. The use of phosphate ion in the dyebath tends to increase the rate of dyeing by approximately 33%.

The application and choice of reducing agent appear to be significantly tied up with oxidation-reduction potentails as well as with the ability to produce free acid from hydrolysis of the reducing agent immediately after addition of copper salts.

It has been demonstrated that, with these techniques, it is possible to dye Orlon, Type 41, in a wide range of colors and to have good wash fastness, crock fastness, light fastness (as tested in direct sunlight similar to that of the Florida test), perspiration fastness and carbonizing fastness.

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WNE Annual Meeting to Feature Panel Discussion

THE Western New England Section, at its Annual Meeting and Election of Officers on October 3, will undertake a panel discussion on "The Dyeing of Synthetic Fibers". The panel will consist of four outstanding representatives in this field: William A Holst, Jr (National Aniline Division); Paul L Meunier (E I duPont de Nemours & Co, Inc); Paul J Choquette (General Dyestuff Corp); and a representative of the Calco Chemical Division.

Arrangements for the program are under the direction of J Edward Lynn, WNE Section Program Chairman.

Rapp's Restaurant, Shelton, Conn, has been selected as the site for the meeting, which is set to begin at 6:30 pm.

Bleaching——

(Concluded from Page P577)

spun fibers respond better than filament fibers, especially when they are blended with natural fibers. It is possible that something new will have to be developed for the bleaching of these synthetics, as has been the case in their dyeing.

This outline of the development of hydrogen-peroxide bleaching has not dwelt on the griefs, troubles of various kinds and discouragements that accompanied each progressive step. The usual resistance to change, reluctance to alter established routine, fear of assuming risk, and comfort in retaining past methods were in evidence most of the time. However, there always appeared a few brave people who were willing to go along on the path of progress.

No one questions the present value of hydrogen-peroxide bleaching, and its being taken for granted is a strong tribute to the pioneers. The work and effort put forth in accomplishing these developments may well be forgotten if the acceptance of the procedures now in use will be the basis of continuing change and improvement in this period of expanding progress in textile processing.

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1953 Meetings Dates, Mid-West Section

THE following meeting dates for 1953 have been announced by Victor H Lawrence, Secretary of the Mid-West Section:

February 14th—Hotel Bismarck, Chicago, Ill

April 18th—Netherland Plaza, Cincinnati, Ohio

June 20th—(Annual Outing) Lake Delavan, Wis

November 7th—Hotel Bismarck, Chicago, Ill

Hudson-Mohawk Section ——

APPLICATION OF PEROXYGEN COMPOUNDS IN THE TEXTILE INDUSTRY*

R A SECORD

Electrochemicals Department
E I du Pont de Nemours & Co, Inc

INTRODUCTION

DILUTE solutions of hydrogen peroxide have been used commercially for nearly a century, but concentrated solutions were not produced in this country until about 1925. Now, 35-50% hydrogen peroxide solutions are common commodities, with U S sales estimated at 60 million pounds a year (calculated as 35%). About two-thirds of this quantity is used in the textile industry where hydrogen peroxide has been established as the preferred bleaching agent. Cotton, wool, silk, synthetic, and miscellaneous animal and vegetable fibers can be bleached with peroxide to good, retentive whites with minimum damage to their physical or chemical structure. Peroxygen compounds are used for oxidizing dyestuffs.

PEROXYGEN COMPOUNDS

HYDROGEN PEROXIDE —— Several peroxygen compounds are used for textile processing, but the most common is hydrogen peroxide. It is available in water-white, extremely stable solutions. The commercial product in general use is 35% hydrogen peroxide. This is equivalent to 130-volume, which means that 1 ml of the solution at 20°C and 760 mm pressure liberates 130 ml of oxygen measured at 0°C and 760 mm pressure. It is slightly acid and weighs 9.4 pounds per gallon.

SODIUM PERBORATE TETRAHYDRATE —— Sodium perborate, another member of the peroxygen family, is widely used both as a bleaching agent and as an oxidizing agent for dyestuffs. Sodium perborate, with a formula corresponding to $NaBO_2.H_2O_2.3H_2O$, is a white powder containing 10% active oxygen. It is stable at ordinary temperatures but begins to give up water of crystallization if exposed to temperatures above 104°F(40°C). Its solubility in water is 2.55 g per 100 ml at 15.5°C, but in acid solution it is more readily soluble. When dissolved in water, oxygen is immediately available, and the solution has all the characteristic reactions of an alkaline hydrogen-peroxide solution with a pH of 10.0-10.2. When heated above 104°F, perborate solutions rapidly liberate active oxygen.

R A Secord

A peroxide expert presents a most useful précis concerning the properties and uses of those peroxygen compounds that are of most interest to the textile chemist, namely, hydrogen peroxide, sodium perborate tetrahydrate, sodium borate perhydrate, and sodium peroxide. Not only are the physical and chemical properties of these given briefly, but the chemical equivalent values of one with another.

The uses of peroxygen compounds in bleaching all common fibers in the forms of knit goods and piece goods, batch-wise or continuously are explained, and practical working formulas for different conditions are given. The oxidation of dyed and printed vat dyes closes the discussion.

The working directions given by the author will prove convenient for ready reference by the chemist who may be called upon to employ peroxygen compounds.

SODIUM BORATE PERHYDRATE—— Another perborate product, "Perdox" sodium borate perhydrate, has recently been introduced. It has a formula corresponding to $NaBO_2.H_2O_2.H_2O$. It is a white powder containing 15.5% active oxygen and is suitable in most cases where sodium perborate tetrahydrate is used. When "Perdox" is dissolved in water, oxygen becomes available, and the solution has all of the characteristics of alkaline hydrogen peroxide at pH 9-10.

SODIUM PEROXIDE —— Sodium peroxide is a yellow powder or granular material having the chemical formula Na_2O_2. It dissolves readily in water and forms an alkaline peroxide solution. It is stable at ordinary temperatures when kept dry or in moisture-resistant containers. Sodium peroxide is a vigorous oxidizing agent and liberates approximately 20% of its weight as active oxygen.

MATERIALS FOR HANDLING

Successful use of peroxide and perborate solutions in chemical applications depends partly upon the selection of proper structural materials for process equipment. The use of improper materials results in corrosion of equipment and wasteful decomposition of peroxide. Such decomposition may be inhibited by adding certain stabilizers to the solution. A stabilizer, to be effective, must deactivate any catalytic materials and continue to function for the life of the peroxide. Corrosion and stability are influenced also by the pH and temperature of solution. To insure satisfactory performance of hydrogen peroxide solutions, care must be taken to prevent contamination with dirt or other foreign materials which may catalyze the decomposition of the peroxide solution. The choice of structural and storage materials that come in contact with peroxide must be considered as a separate problem for each specific application.

In general, it is recommended that alkaline hydrogen-peroxide solutions in bleaching strengths be handled in stainless-steel equipment. Other satisfactory materials are

* Presented before the Hudson - Mohawk Section at Jack's Restaurant, Albany, N Y, on March 21, 1952.

wood, special rubbers, glassware, earthenware, and enamel. Iron, steel, nickel, and lead give good performance under certain conditions, particularly when the hydrogen-peroxide solutions contain an adequate amount of sodium silicate. Copper and brass are not satisfactory.

Acid solutions, such as those used for bleaching wool, can be satisfactorily handled in glassware, earthenware, enamelware, tin, lead, special rubbers, and certain types of stainless steel.

Commercial hydrogen peroxide can be satisfactorily stored in original aluminum drums or glass carboys, or in aluminum tanks. Pipe lines for conveying the peroxide from the container to the point of use can be made from commercial 3S aluminum. Where pumps are used, the parts which come in contact with peroxide must be stainless steel or equivalent peroxide-resistant alloys, such as "Worthite". Tygon is a satisfactory gasket material.

Sodium peroxide can be stored only in the original containers. It can be handled in iron, steel, glass, enamelware, or earthenware containers, but should never be transported in paper or other combustible containers. Although it must be kept away from combustible materials, there are no difficulties in handling if the prescribed safety rules are followed. If spilled on the floor or any surface it should be flushed immediately with a large amount of water.

EQUIVALENT VALUES

The peroxygen compounds that have been described are very common in dye houses and bleacheries. Before outlining specific uses for these materials it is perhaps advisable to point out the equivalent values of each.

It requires 7.3 pounds, or 3.1 quarts, of hydrogen peroxide 35%, which contains approximately 16.5% active oxygen, to produce 100 gallons of a 1.0-volume bleaching solution. A bath of the same strength can be prepared by using 6 pounds of sodium peroxide per 100 gallons of water. This 1.0-volume solution also has approximately 6 pounds of sodium hydroxide present, which makes it attractive where a caustic solution is desired. Sometimes it is advisable to use a combination of hydrogen peroxide and sodium peroxide to prepare a bleaching mixture of specific strength. This eliminates the necessity of adding caustic soda.

Twelve pounds of sodium perborate tetrahydrate per 100 gallons is required to produce a 1.0-volume bath. This material also gives an alkaline solution when dissolved in water, and, in most cases, it will not be necessary to employ added alkali. The resultant mixture is buffered at approximately pH 10.0-10.2. A similar solution with pH 9.6-10 can be prepared with 7.8 pounds of "Perdox" sodium borate perhydrate per 100 gallons.

In stating the amounts necessary to produce a bath having a common volume concentration, the value of 1.0-volume was used merely as an example. The nature of the goods being processed, as well as mill conditions and equipment being used, will determine and dictate both the strength of the bleaching solution and the bleaching agent to be employed.

BLEACHING METHODS USED FOR VARIOUS MATERIALS

COTTON PIECE GOODS —— About 65% of the cotton piece goods bleached in the U S is processed in continuous peroxide bleaching equipment. More than 125 systems of all types have been installed and are now in operation. Although the exact total productive capacity is difficult to calculate, it is estimated that the existing ranges could bleach 170 million yards a week, or 9 billion yards a year, of 4-yard print cloth if operated at an average speed of 200 yards a minute, 120 hours a week.

The speed of any continuous bleaching system is limited by the capacity of the intermediate washers. Productive rates of approximately 300 yards a minute are possible with high-speed washers of the newest design. A typical du Pont rope system requires the following basic equipment:

Preliminary Washer
Caustic Saturator
Caustic J Box
Two Intermediate Washers
Peroxide Saturator
Peroxide J Box
Final Washer

In addition to this basic equipment, auxiliary equipment, such as variable speed drives, control instruments, mixing tanks, etc, is required.

When handling fabrics to be dyed after bleaching or to be finished white, the sequence of operations is as follows:

1) The cloth, which has previously been singed and desized or grey-soured, is washed in the first washer.

2) The cloth leaving the first washer is squeezed to reduce the water content to about 80%.

3) The goods are passed into the rope saturator, which contains a 3-4% solution of caustic soda. If the cloth being processed contains colored yarns, a ¾% alkaline peroxide solution (based on 35% hydrogen peroxide) is used instead of the caustic-soda solution. As the cloth leaves the saturator, it is squeezed to about 100% moisture content.

4) The chemically saturated cloth next passes through the heater tube, where the temperature is raised to just under 212°F.

5) The saturated, heated cloth next enters the J box proper, where a mechanical piling device plaits it down in the box in a pattern designed to prevent tangling and facilitate removal at high speeds. The cloth remains in the J box for approximately 1 hour.

6) The scoured cloth is pulled out through two intermediate washers in which the residual caustic soda is reduced to below 0.2%. It is then ready for bleaching.

7) Before cloth enters the peroxide saturator, the moisture content is again reduced to about 80% by passage through rubber squeeze rolls.

8) The cloth enters the peroxide saturator, which contains an alkaline peroxide solution with a pH of approximately 10.6 and with a peroxide concentration of about ¾% (based on 35% hydrogen peroxide). The cloth is again squeezed to a moisture content of 100%.

9) The peroxide-saturated cloth then passes through a second heater tube, where the temperature is raised to 210°F. Colored-yarn fabrics are sometimes bleached at lower temperatures.

10) The cloth is next stored in a J box for one hour, during which time bleaching is completed. A small amount of residual peroxide remains in the cloth along with by-products of the bleaching reaction and some unreacted alkali.

11) The cloth then receives a final wash. Only one washer is required since the material is relatively free from impurities.

Before the continuous method of bleaching cotton piece goods was introduced, the kier method of bleaching was quite widely used. It is still used in mills where the production requirement is too limited to justify a continuous system. Next to continuous bleaching, this is considered the most economical method now in use. In the kier method, cotton piece goods are boiled out in a solution containing about 3% caustic, based on the weight of the goods, then bleached with about ½ to 1½% hydrogen peroxide 35%, 1-3% silicate, and a wetting agent. A ratio of about 50 gallons of bleach liquor to 100 pounds of goods is used; this makes it possible to obtain a peroxide concentration that produces a good bleach in 4-8 hours, depending on the type and weight of goods being treated. In some cases it may be desirable to introduce additional alkali by substituting sodium peroxide for some of the hydrogen peroxide. This may shorten the bleaching time and improve the resultant whiteness.

When colored fabrics are bleached, the caustic boil-off is eliminated. After being desized, colored material is given a scour with silicate and a small amount of peroxide, then bleached in a low-concentration peroxide bath.

COTTON KNIT GOODS——A number of continuous systems based on the du Pont process are being used for processing cotton knit goods. Production per unit is running as high as 100,000 pounds per week and can be increased if necessary. The finished fabric is commercially acceptable and compares favorably with that produced in the best batch bleaching processes.

Experience has shown that most knit goods can be bleached rapidly and economically to a full white in a single run through a J box, although heavy goods may require two treatments.

The sequence of operation with knit goods is as follows:

1) Wet out with a suitable wetting agent.
2) Wash.
3) Saturate with alkaline peroxide (3% based on 35% hydrogen peroxide, pH 10.6-10.8).
4) Squeeze to 100% moisture content.
5) Heat to operating temperature (200-211°F) and store in J box for one hour.
6) Wash.
7) Extract or squeeze.
8) Dry.

When processed in a kier, cotton knit goods are usually bleached in a one-step process, which combines the boil-off and bleach operation. In this process the rolls of knit goods are tied or sewed together and wet out through a standard slack washer in warm water containing the necessary amount of the desired wetting agent. It is customary to wet out and load the kier simultaneously. Piling should be done from the top of the kier; the operator should never enter the kier. After the kier has been filled, the peroxide bleach solution is added. The pump is then started and the cold bleach bath is circulated through the goods for 30 minutes. The steam is turned on and the temperature gradually raised to 180°F over a period of 2-3 hours. Bleaching is continued for 5-6 hours at 180°F. After bleaching is completed, the kier is drained and refilled with warm water, which is circulated for one hour. This rinse is repeated, and finally a cold wash is given. In most cases the cloth is pulled out of the kier through first warm and then cold water. It is then squeezed or extracted and dried.

Cotton knit goods are frequently bleached in slack washers or dye tubs. In such processes, the tub is first filled with about 800 gallons of water for 1,000 pounds of cloth. Next, the machine is loaded, about 3 rolls or 60-100 pounds in each compartment, the total batch being about 1,000 pounds.

With the machine running slowly, 30 pounds of sodium peroxide or 30 pounds of 35% hydrogen peroxide and 30 pounds of caustic soda are added to the water. The machine is run cold for 15 minutes, and then the temperature is raised to 180°F. It is customary to bleach for 1 hour at 180°F before raising temperature finally to 200°F for ½ hour.

The tub is then drained and the cloth is washed thoroughly. If only a partial bleach is required, the cloth is dried and finished. If a full white is desired, the machine is again filled with water and 25-50 pounds of sodium hypochlorite and 10-20 pounds of sodium silicate are added. The cloth is again run cold for 15 minutes, the temperature is raised to 120°F and bleaching is continued for ½ hour. Next the temperature is raised to 150°F, and 10-20 pounds of sodium bisulfite to neutralize the residual hypochlorite is added. Lastly, the cloth is washed, blued to shade, and finished.

RAYON AND ACETATE PIECE GOODS——Reels, jigs and becks are used for bleaching piece goods woven from synthetic yarns, such as rayons and acetates. Although the liquor ratios employed are relatively high, economical peroxygen formulas can usually be developed.

A preliminary boil-off or scour is applied in order to remove soil, mineral oil, and graphite stains. If the goods contain a fugitive dye for marking the twist and weaving, it may be necessary to strip with a sodium hydrosulfite or sodium sulfoxylate formaldehyde. These materials must be used carefully since, in equipment containing brass or monel metal, the sulfides formed may undo all the good effected and cause stains or discolorations to appear when the material is bleached.

A number of mills have continuous boil-off machines, and some open-width continuous equipment for handling rayon has recently been put into operation. A shorter scouring time with better results is claimed for these ranges. After a preliminary scour the goods are usually strung up in slack reel machines for bleaching. Sodium perborate is widely used in this operation. The ratio of liquor to goods varies considerably with the size and type of reel machine being used. However, on a large machine in which 50 pieces or approximately 1,000 pounds of goods can be handled, 3% sodium perborate, based on cloth weight, is sufficient to produce a good bleach in ½ hour. This concentration is equivalent to approximately 2½ pounds of sodium perborate per 100 gallons of bath. At operating temperature the solution has a pH of about 10.4. Since there is a temperature differential of about 20° F from front to back of the tub, the temperature should be maintained between 160 and 170° F in the front to get efficient bleaching throughout the bath. Laboratory tests on acetate fabrics bleached in this manner indicated that very little saponification of the yarn had taken place.

Rayon sateens, drills, twills, and materials of similar construction are scoured and bleached on jigs. These goods are often heavily sized and must be given a treatment with an enzyme before scouring. After the usual scour and 2 ends in warm water, the goods are ready for bleaching. A bath containing 7-8 pounds of 35% hydrogen peroxide and 2-3 pounds of sodium silicate per 100 gallons may be used. Usually, a satisfactory bleach is obtained with 6-8 ends at a temperature of 180° F. If the material contains acetate yarn, the bleaching temperature should not exceed 160° F.

In some cases satisfactory results can be produced by combining the scour and bleach in one operation. For this the bath should contain about 4 pounds of mild soap, 6 pounds of sodium silicate, and 9-10 pounds of 35% hydrogen peroxide per 100 gallons of solution. The treatment should be carried out for about 1½ hours at 180° F.

SILK——The concentration of hydrogen peroxide used in bleaching silk piece goods that have been degummed depends on the volume and variety of fabrics handled by the mill. In cases where light, sheer goods are segregated, a bath of relatively low concentration can be maintained. In mills where materials of varied weights and construction are handled in the same tub, volume concentrations ranging from 0.5-1.5 are used for bleaching. The time of immersion varies with the weight and construction of the goods being bleached. As a general rule the bleaching temperature is not higher than 180° F, and the silicate content of the bath does not exceed 6 pounds per 100 gallons. These conditions are the same whether the material is being bleached in the rope form, in a reel machine, or as skeins in an open tub or beck.

WOOL——Wool is bleached in various forms but usually as raw stock, yarn, or piece goods. In each of these three classifications the wool contains different amounts and kinds of impurities. Wool raw stock contains natural impurities as well as dirt, burrs, etc. In the continuous bleaching of raw stock the actual bleaching is conducted in a bowl placed at the end of the wool-scouring train. The wool enters the bowl from the last rinse and passes through a dilute, slightly acid solution of hydrogen peroxide. Usually the train is so designed that the wool remains in the peroxide bowl for 2-3 minutes. At the exit end of the bowl it passes through squeeze rolls and is then dried without rinsing.

The bleach bowl must be lined with a material that will not be affected by or that will not catalyze the decomposition of the acidified peroxide solution. In most cases, stainless-steel or lead-lined bleach bowls are used, and the racks are of stainless steel.

The wool enters the bleach bowl carrying some alkali from the scouring operation. It is necessary to feed an acid, usually sulfuric, to neutralize the alkali and to maintain a pH of 3.5-5.5 in the bleaching bath. The concentration of the peroxide bleach varies from 0.25 to 2.5 volume, depending upon the degree of bleaching required.

Wool yarn and wool hosiery are generally bleached in standing baths. Previous to the bleaching operation the wool is well scoured by usual methods. The actual bleaching may be done in wood, stainless steel, tile, or transite vats. The vat should be fitted with a perforated or slatted false bottom placed about 8 inches off the bottom and also with perforated or slatted grates to be placed on top of the yarn to keep it completely submerged during the bleaching operation. In constructing the wood grates and false bottoms, the wood must be fastened by materials that will not cause trouble in bleaching. Wooden pegs or stainless steel screws or bolts are satisfactory; iron, copper, or brass cannot be used. Some means must be provided to hold the top grate under the surface of the liquor.

The bath is heated by a closed steam coil located beneath the false bottom of the bleach vat. This coil may be of lead, stainless steel, or aluminum, the choice depending upon the alkali to be used in the formula. Lead is satisfactory if the formula contains silicates and less than 50% of the total alkali as pyrophosphate.

The bleach bath is prepared by filling the vat with water, heating to desired temperature and adding 2-3 pounds of sodium silicate or 1½-2 pounds of anhydrous tetrasodium pyrophosphate and 10-20 pounds of 35% hydrogen peroxide per 100 gallons. The scoured yarn is well rinsed and extracted, and then added to the tank in a head. Hosiery may be entered loose or, if sizes are to be kept separate, in net bags or stainless-steel baskets. When the vat is loaded, a rack is placed in position to submerge the goods. The yarn is allowed to bleach for 5 or more hours and then is rinsed and dried.

There are various procedures for bleaching woolen goods. For the best white they may be submerged in a standing bath similar to that described previously. The desired shade may sometimes be obtained by bleaching in a reel machine for 2-3 hours at a temperature of 120-125° F. The quantity of peroxide required depends upon the white desired. After the desired shade is obtained, the peroxide solution may be pumped to a storage tank and replenished for subsequent bleaching operations. The bleached material may then be rinsed free of all chemicals and dried in the usual manner.

Recent research work has indicated that it is possible to pretreat wool with formaldehyde and then to subject the goods to a higher bleaching temperature or stronger bath to shorten the over-all time of bleaching. It is expected that this process will permit more rapid bleaching of wool with no more damage to fibers than normally occurs in the regular procedure. Mill-scale trials have been planned for evaluating this new method.

COTTON YARN——The peroxide bleaching of cotton yarns in package machines is rapidly replacing older bleaching methods. Many variations in formula are possible, the following being only one of many which was worked out very successfully in stainless-steel equipment.

A typical method is to load the packages in the machine, to add water to the proper level and then to add ½-2% of a good wetting agent, 2% of soda ash, and 4% of silicate. The liquor is circulated for about 20 minutes, during which time the temperature is raised to 120° F. Then 4-6% hydrogen peroxide 35%, based on the dry weight of the yarn, is added and the temperature raised to 180° F. The yarn is bleached from 2-4 hours at this temperature, then thoroughly rinsed and blued if desired. The packages are hydro-extracted and dried in a warm current of air.

MINOR VEGETABLE FIBERS——There are many minor vegetable fibers, such as hemp, jute, sisal and straw, which are used in commercial quantity. Many of these fibers are used in their natural state, while others are bleached, partially bleached, dyed or finished in some other manner to improve their appearance and adapt them for novelty uses. There are wide variations in the natural colors, quality, luster, and strength of these fibers, and in many cases the fibers are weakened or even destroyed if any attempt is made to process them.

Processing of vegetable fibers may consist of only bleaching and washing, or the fiber may be given a preliminary scour and wash before bleaching. In some cases it has been found that successive bleaches at lower temperatures and chemical concentrations give better results than a single bleach. Some vegetable fibers are weakened by bleaching, while others cannot be bleached to a full white without undergoing structural disintegration.

In general, it is advisable to proceed cautiously when attempting to develop a bleaching procedure for most vegetable fibers. Mild conditions, such as low peroxide concentration, weak alkalinity, and only moderate temperatures, are recommended unless it has been established that the material under test will withstand more severe conditions.

OXIDATION OF DYESTUFFS

Peroxides are now being used extensively for the oxidation of dyestuffs. Sodium perborate is ideally suited for the oxidation step necessary to fix vat dyes. Hydrogen peroxide can also be used to good advantage. A dilute solution of perborate is sufficient to insolubilize the impregnated dyestuff. Oxygen from the perborate or peroxide reacts with the dyestuff, reoxidizing it to the insoluble color compound. The amount of perborate required is approximately 1% based on the weight of the goods being dyed. This concentration varies somewhat with different conditions since some dyestuffs oxidize more slowly than others. The necessary amount can be determined experimentally. For cotton or rayon piece goods dyed on a jig, 2-4 ends through a dilute peroxide solution at a temperature of 120° F gives a good oxidation. Pad-jig work is treated in the same manner. Vat colors dyed on silk are oxidized in a similar manner, although it may be advantageous to add a small amount of acetic acid to the fixing bath.

On cotton or rayon either printed or dyed vat colors may be fixed by passing the goods through a bath containing 5-8 pounds of sodium perborate per 100 gallons of solution maintained at 120° F. Vat dyes on printed silks are fixed by treating the fabric in a bath containing 3 pounds of sodium perborate and 4 pints of acetic acid per 100 gallons of solution at 120° F.

Both hydrogen peroxide and sodium perborate, when used properly, will give satisfactory results in oxidizing dyestuffs.

SUMMARY

Peroxygen compounds are recognized as versatile bleaching agents and can be adapted to fit most commercial bleaching processes. When the proper combination of time, temperature, pH, and concentration are used, peroxides are economically competitive with other bleaching chemicals. Only simple control tests are required throughout the bleaching cycle, and a formula can usually be developed to use existing equipment. The wide-spread acceptance of peroxides is primarily due to the ease of handling and the high quality of bleach produced. Peroxides give full whites with excellent color stability and with minimum loss in tensile strength.

ABSTRACTS

The Coloring of Aluminum

W W G Hübner, *Ciba Rev* 92, 3321-4, June, 1952.

A prerequisite for the coloring of aluminum is a well-formed oxide coating. When aluminum is used as the anode in a suitable electrolyte, it becomes covered with an oxide film (hence the name, anodic oxidation). Such coatings consist mainly of aluminum oxide covered with hydroxide. This hydrated oxide penetrates well into the pores of the metal.

The anodic oxide film with its porous structure resembles textile fibers in many respects and, like them, may be dyed with aniline colors. As is well known, dyestuffs, which are usually applied in an aqueous solution, either combine with the textile fiber or are simply adsorbed in a purely mechanical way. Similarly, in aluminum coloring, a chemical process takes place with a large number of coal tar dyes, while others are only superficially adsorbed.

The coloring of aluminum with inorganic salts is particularly suitable for constructional material. Yellow or "gold" shades are usually obtained by immersion in a solution of ferric ammonium oxalate at 150° F. The resulting shade varies from the lightest yellow to the darkest red gold tone. A treatment with cobalt acetate followed by potassium permanganate will produce "old brass" shades.

Coloring in organic dyebaths is considerably simpler and offers unlimited possibilities. Preliminary anodizing itself requires less time and trouble. In a 20 per cent sulfuric acid bath with a current density of 1.5-2 amps per sq dm, 30 minutes will in most cases be enough for the anodic treatment at 68° F. Only if the film has to be dyed black and is expected to resist sunlight and weathering does longer treatment become necessary.

The dye liquor, which must be operated at the required pH value, is kept in containers of rustless steel or aluminum, preferably lined with foils of plastic material or treated with synthetic varnishes. One after the other, the objects are dipped into the dye liquor and left there until they have assumed the required depth of shade. The normal operating temperature is 150° F. Before dyeing, the anodized films are well rinsed and immersed in cold nitric acid, then rinsed in cold water, and dipped in the dye liquor while still wet.

Dyeing is followed by "sealing", a process which closes the pores and gives the surface its final smoothness. This operation is preferably done in gently boiling water or with hydrolyzing salts (nickel-cobalt acetate and Glauber's salt are suitable). In certain cases, oil and wax sealing is employed. The dried surfaces are finally treated with lacquers and baked.—WHC

The Continuous Dyeing of Vat Dyes: the Standfast Molten Metal Technique

J Ardon, M R Fox, and R W Speke, *J Soc Dyers & Colourists* 68, 249-56, July, 1952.

The authors describe the construction of a laboratory model of the Standfast Molten Metal machine which was used to evaluate this new dyeing technique. The laboratory machine has a stainless steel trough 6″ wide, 30″ high, and 2″ thick, filled with Woods metal, and divided into two compartments by a central fixed plate running almost to the bottom. The whole is immersed in a boiling water bath to maintain the metal alloy at the necessary temperature. The two compartments have a capacity of 300 cc above the molten metal; dye solution is added to one side and salt solution to the other.

The fabric, 5″ wide, after being preheated, passes downward through the reduced vat dye liquor at 80° C, and has a 50-inch passage through the molten alloy, going down one side, around a roller, and up the other side. It emerges through the salt solution, is squeezed, and is then oxidized, rinsed, and soaped.

Using this apparatus a program of investigations was undertaken with a view to obtaining first-hand information on the factors affecting the application of vat dyes to cellulosic textiles under such conditions. This program is described in detail. Tests were run with (a) the leuco vat dyeing process; (b) pigment padding, drying, and developing through the molten metal machine; (c) pigment padding and developing wet through the machine.

The authors draw the following conclusions (which they emphasize are based on *laboratory* apparatus):

The leuco dyeing process is technically an ideal continuous vat dyeing method yielding results equal, and in many respects superior, to conventional jig dyeings, with the minimum of operational difficulties. Its only limitation lies in the dyeing of heavy or tightly woven fabrics, where difficulties are encountered in obtaining adequate dye penetration into the fabric structure. The latter difficulty can be overcome by prior pigment padding followed, with or without intermediate drying, by development through the molten metal machine. The laboratory evaluation of this sequence has shown that full reduction and fixation of the vat dye, in the limited time of immersion in the molten alloy, are difficult to achieve in certain instances. By modification of the composition of the pad liquor and the reducing feed liquor coupled with dye selection, however, adequate reduction and fixation of the padded dye can be achieved with approximately 6-6.5 seconds in the molten alloy at 95-100° C.—WHC

Investigations into the Effect of Carbonizing Processes on Nylon Yarn and Fabric

(from Technical Service Manual of British Nylon Spinners Ltd)

Textile Mercury & Argus 127, 194-5, August 1, 1952.

In principle, carbonizing is the conversion of cellulose into hydrocellulose by treatment with acids (either sulfuric acid solution or hydrochloric acid gas) or acid-producing salts (such as aluminum chloride) followed by drying and baking.

Trials on both a laboratory and works scale have been carried out, in the former case using sulfuric acid and in the latter case using sulfuric acid and hydrochloric acid gas as carbonizing agents, since these are most commonly used.

The trials included an assessment of physical and chemical degradation of the nylon and the effect on its dyeing properties. For this purpose the nylon component was continuous filament in both yarn and fabric form. The fabrics tested included nylon satin, stitched to a length of woolen cloth, and a fabric woven from a twist of worsted and nylon in both warp and filling.

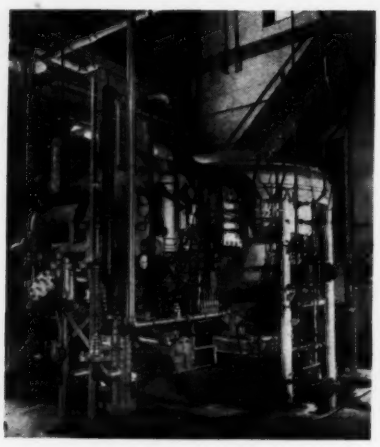
The following conclusions were reached:

a. Nylon yarn will satisfactorily withstand the normal sulfuric acid carbonizing processes used for wool piece-goods. It will not withstand rag carbonizing processes using hydrochloric acid gas; the latter causes marked degradation, but not total destruction of the nylon material.

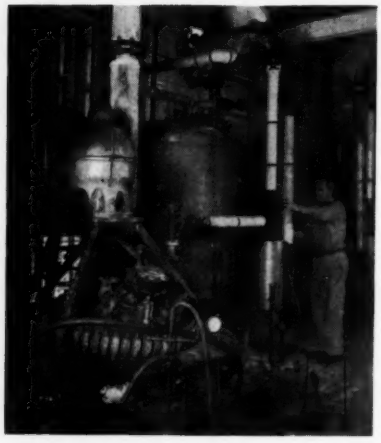
b. When nylon is used as a core yarn, or is folded or blended with wool, the effects of normal piece-goods carbonizing processes on the nylon component are probably minimized due to protection by the wool—the degree of protection depending upon the percentage composition of the mixture, and yarn and fabric construction.

c. When nylon yarns and fabrics are given a normal sulfuric acid carbonizing treatment, it is found that the dyeing properties of the nylon, using acid, premetalized or chrome dyes, are for most practical purposes relatively unchanged.—WHC

NEWS



Molecular Still



Esterification Unit

• Arnold, Hoffman Expands Harkness & Cowing Division

Arnold, Hoffman & Co, Inc, with headquarters in Providence, Rhode Island, recently completed a series of major expansion moves at their Harkness & Cowing Division plant in Cincinnati, Ohio.

Among the important equipment additions to the Harkness & Cowing plant is the new illustrated molecular still, manufactured and installed by Distillation Products Industries, a division of Eastman-Kodak. A completely new esterification unit has also been installed, as illustrated.

The expansion is said to have come as a direct result of an earlier Arnold, Hoffman decision to move production of synthetic products adjacent to the basic raw materials production at the Harkness & Cowing Cincinnati Division. Thus, Harkness & Cowing, which was originally acquired by Ahco because of their prime position as producers of fatty acids, has become the key plant in the parent firm's production of synthetic products, which include distilled esters, distilled oleic acid, the Ahcovel line of substantive softeners, and wetting agents. The active expansion of vat dyestuff facilities in Arnold, Hoffman's Dighton, Massachusetts plant, which formerly housed the synthetic products manufacturing facilities, was another substantial reason for combining synthetic products production with the production of basic raw materials for these products in Cincinnati, it is reported.

• Revised Set-up Between Diamond Alkali, Kolker

Diamond Alkali Company has announced a revision in the administration setup of its subsidiary, Kolker Chemical Works, Inc, Newark, N J, and Houston, Texas.

Leon A Kolker, founder and president of the Kolker Company, retired on August 31. The activities of the Kolker concern continue to be carried on by J G Brunton, in charge of Kolker sales, and Charles H Kolker, in charge of operations and engineering. The functions of these two men are now coordinated under A L Geisinger, Diamond Alkali vice president in charge of the Organic Chemicals Division.

• Italian Exhibition Attracts International Interest

The Second Annual Cotton, Rayon and Textile Machinery Exhibition, to be held in Busto Arsizio, Italy, from September 27th to October 12th, is reportedly attracting widespread interest among manufacturers of textile machinery, textile chemicals and accessories throughout the world.

The main purpose of the show is to place at the disposal of all textile and machine manufacturers an efficient means to foster their reciprocal business connections and to establish profitable business relationships with Italian and other European markets.

• Sixth N E Conference on Quality Control

The Sixth N E Conference on Quality Control will feature complete textile panels on October 2 and 3 at the Hotel Sheraton in Worcester, Mass. Although in previous years the papers delivered at these Conferences have been of a general nature, this year the needs of the textile industry in particular are being filled by the addition of these talks on applications of quality control techniques to textile manufacturing.

At least two such panels have been planned — one by Edward Rudnick of Wamsutta Mills, New Bedford, Mass, and one by Robert Jones of Bigelow-Sanford, Amsterdam, New York. Also, the exhibits at the Conference will show how quality control techniques have been applied in particular companies. Textile organizations are included in the list of exhibitors.

The planning of the textile panels as part of this N E Conference follows the formation of the Textile Division as a separate industry-wide entity of the American Society for Quality Control at Clemson, S C in August. All textile people interested in quality control are welcome to attend this Conference. For further information, contact W Adam, Bryant Electric Co, Bridgeport, Connecticut.

• Carbide Reduces Prices On 'Tergitol' Wetting Agent 7

A price reduction of six cents per pound on "Tergitol" wetting agent 7, has been announced by Carbide and Carbon Chemicals Company, a Division of Union Carbide and Carbon Corporation. The new prices are:

Eastern Territory (East of Idaho, Utah and Arizona)

37.0c per pound in less than carload lots in drums

36.0c per pound in carload lots in drums

Western Territory (West of Montana, Wyoming, Colorado, and New Mexico)

39.0c per pound in less than carload lots in drums

38.0c per pound in carload lots in drums

All l c l and c l shipments are in nonreturnable drums, f o b delivery point of rail carrier nearest point of destination.

• Bancroft Approves Use of Homogesols in Production Of Everglaze Fabrics

Joseph Bancroft & Sons Co of Wilmington, Delaware in a letter signed by Arnold Lippert, Vice President of Research and Development, reportedly have approved the use of Homogesol P, SP and LTD in the production of effects which may be labeled "Everglaze." The letter states in part that the urea-formaldehyde shrink proof and crease proof resins manufactured by the Woonsocket Color & Chemical Co "may be satisfactorily used in the production of 'Everglaze' effects."

Homogesol P and SP are shrink proof and crease proof urea-formaldehyde cream resins and Homogesol LTD is the syrup resin which Woonsocket Color & Chemical is currently manufacturing. In addition to these, the Company's Research Department is currently working on the development of several different resin finishes, some of which are now receiving practical trial in a number of mills.

• Alcolac Opens New Sales Districts

The American Alcolac Corporation, 3440 Fairfield Rd, Baltimore, Md, producers of fatty alcohol sulfate detergents, recently announced the establishment of two national sales districts. The New York Office will be managed by Wiley J Pickett while the Chicago office, at 6 North Michigan Avenue, will be directed by Alfred A Michaud. The Divisional Sales Managers will report directly to the Corporation's main office at Baltimore, Md.

Distribution in Los Angeles continues to be handled by the Braun Corporation.

In another move to expand facilities the Corporation's Canadian branch has opened new offices and warehouse facilities at 937 Victoria Square, Montreal.

• Sales of Laboratory Materials Climb

Sales of apparatus, instruments, and supplies for the nation's scientific research, development and industrial control laboratories continue to climb according to the Mid-Year sales report of the Laboratory Apparatus and Optical Instrument Sections of the Scientific Apparatus Makers Association.

The report shows that sales of laboratory apparatus for the first half of 1952 were $61,811,924 for the 122 companies which are members of this Section of the industry's national organization. This is 7.5 per cent over sales for the corresponding period last year.

Optical instrument sales for first-half 1952 were $6,795,199, or 15.8 per cent over last year. There are 12 companies in this Section.

Because they sell largely to the research and development laboratories of industry, the Government and educational institutions of the nation, sales figures for these companies generally serve as a barometer of the nation's research and development activities, according to Kenneth Andersen, executive vice president of the Association.

Although increases are sizeable (average 8.25 per cent for the two Sections) they may indicate a leveling out of the curve which has been rising rapidly since the start of war in Korea June 25, 1950. Sales rose 10 per cent in 1950 over 1949. The next year they were 27.4 per cent over 1950.

• Hooker Opening Chicago Sales Office

Hooker Electrochemical Company is opening a new sales office at 1 North La Salle Street, Chicago, according to an announcement by R L Murray, President. Charles Y Cain, Hooker's sales representative in the Chicago area since 1945, has been named district sales manager, and Donald McKechnie, who has been in the company's main office for several years, will be Chicago office manager.

The new $12 million Hooker caustic soda chlorine plant now under construction at Montague, Michigan, near White Lake, will reportedly be the closest producing plant of its kind to the Midwest market.

• Federal Resinous Chemicals Formed

The formation of Federal Resinous Chemicals, Inc, 210 Wythe Ave, Brooklyn, N Y, has been jointly announced by Federal Adhesives Corp, Federal Latex Corp, Resinous Chemical Mfg Co, and Adex Mfg Co.

The new Corporation will specialize in the production of textile processing chemicals, such as synthetic resins, detergents, finishes, etc, it is learned, and will operate from its plant in Brooklyn. Southern territories will reportedly be serviced from another plant in Baltimore, Md.

• Robbins Mills, Hego Fabrics Merge

Karl Robbins, chairman of the board of Robbins Mills, Inc has announced the merger of that company and Hego Fabrics, Inc. The latter organization will reportedly continue as an independent operating unit with the same organization and personnel.

Herman Goodman, founder of Hego Fabrics in 1933, has been elected a director and executive vice president of Robbins Mills, Inc. Before setting up his own business he was, for seven years, vice president of Colonial Mills, Inc, predecessors of Robbins Mills, Inc.

• NETF Fund Campaign Going Well

Melville Weston, General Chairman of the New England Textile Foundation's annual fund-raising campaign, has announced that the 1952 goal is "well in sight with $38,000 in contributions already in hand or pledged". Announcing official commencement of this year's drive on September 1, Mr Weston appealed for consideration from firms in the textile and allied fields throughout the country.

• Celanese Chemical Division Branch Office

The Chemical Division of Celanese Corporation of America has opened a new branch office in Charlotte, N C, from which William Milheim will operate as its sales representative for the Southeastern District, which includes the states of Virginia, North Carolina, South Carolina, Georgia, Florida and Tennessee.

Opening of the new branch office follows closely the establishment of terminal facilities at Rock Hill, South Carolina.

Address of the new Celanese branch office is 1603 Liberty Life Building, Charlotte 2, N C.

• Savage Heads New Diamond Alkali Subsidiaries

S S Savage has been elected president of two new export companies in New York, subsidiaries of the Diamond Alkali Company of Cleveland, Ohio.

The new concerns, both incorporated in Delaware, began operations on September 1. Diamond Alkali Inter-American Corporation will be the sales outlet for Diamond Chemicals in Latin-America and other countries in the Western Hemisphere outside the United States; and Diamond Alkali International, Inc, will serve all other areas of the world.

Offices and facilities of both are at 122 East 42nd St, New York, N Y.

• 'Forecasting the Election' to be Subject at DCAT Business Meeting

Archibald M Crossley, Pres, Crossley, Incorporated, will be the speaker at the 62nd Annual Meeting of the DCAT to be held at Pocono Manor Inn, Pocono Manor, Pa, September 25-28. His subject will be "Forecasting the Election".

Stanley I Clark (VP, Sterling Drug Inc), Chairman of the Business Program Committee, announces that Mr Crossley will be the sole speaker at the business session scheduled for Friday morning, September 26.

At this session the Section conducts the business of its Annual Meeting including the presentation of reports by officers and committee chairmen and the election of a new Executive Committee.

Mr Clark has also scheduled a showing of the film "The 1952 Market—A Preview" by Arno Johnson, Director of Research for J Walter Thompson Co, because of its long range business forecast.

Charles M Macauley (Charles M Macauley & Associates), Chairman of the DCAT, has announced the following chairmen to head committees for the Annual Meeting.

Banquet Committee——Chairman, Lloyd I Volckening (The Ivers-Lee Co), Vice Chairman, Harold F Cummings (Vitamerican Oil Corp); *Business Program Committee* —— Chairman, Stanley I Clark (Sterling Drug Inc), Vice Chairman, William W Huisking (Chas L Huisking & Co, Inc); *Evening Entertainment Committee*——Chairman, Claude A Hanford (Pharaco, Inc), Vice Chairman, Donald S Cushman (Innis, Speiden & Co); *Greetings Committee*——Chairman, Charles P Walker, Jr (Chas Pfizer & Co, Inc).

Golf Committee —— Sydney N Stokes (Merck & Co, Inc), Vice Chairman, Harry C McKenzie (Ortho Pharmaceutical Corp); *Ladies Golf Committee*——Chairlady, Mrs F Dean Hildebrandt; *Ladies Card Party Committee*——Chairman, Ralph A Clark (J T Baker Chemical Co); *Memorials Committee* —— Chairman, Russel J Fosbinder (The Maltbie Labs, Inc).

Miscellaneous Sports Activities —— Chairman, J David Hayden (Gelatin Products Div, R P Scherer Corp), Vice Chairmen, W Boyd O'Connor (Ayerst, McKenna & Harrison, Ltd) and Fred J Stock (Mathieson Chemical Corp); *Sub-Committees*—— *Archery*——Chairman, Fred G Singer (E I du Pont de Nemours & Co, Inc); *Croquet* ——Chairman, H W Blades (Wyeth Incorporated); *Horse Shoes* —— Chairman, J N Conover (L Sonneborn Sons, Inc); *Shuffleboard*——Chairman, P J Cardinal (Hoffmann-La Roche, Inc); *Putting*—— Chairman, R P Neptun (N Y Quinine & Chemical Works).

Publicity Committee——Chairman, Murray Breese (Murray Breese Associates), Vice Chairman, George S McMillan (Bristol-Myers Company; *Publication Committee* —— Chairman, Russell Boland (Topics Publishing Co, Inc); *Committee For General Reception* —— Chairman, James G Flanagan (S B Penick & Co), Vice Chairman, Dudley Dunlop (Mallinckrodt Chemical Works); *Registration Committee* —— Chairman, Hugh S Crosson (McKesson & Robbins, Inc), Vice Chairman, Stephen F Urban (E R Squibb & Sons).

• Rare Chemicals Clearing House

Chemicals prepared in colleges and universities are now being used to facilitate the work of both academic institutions and industry. Chemicals Procurement Company, 550 Fifth Ave, New York 36, N Y, has announced the establishment of a rare chemicals pool with the cooperation of chemistry departments throughout the country. Lists are submitted by these departments to CPC of rare inorganics, metallo-organics and organics that might be stored and forgotten or thrown away.

OBITUARY

HUGH J FRASER

HUGH J FRASER, vice president in general charge of all plant operations in the United States of The International Nickel Company, Inc, died at St Mary's Hospital, Montreal, Quebec, on August 22 after a brief illness. He was in his 55th year. His home was at 45 Woodbine Avenue, Larchmont, New York.

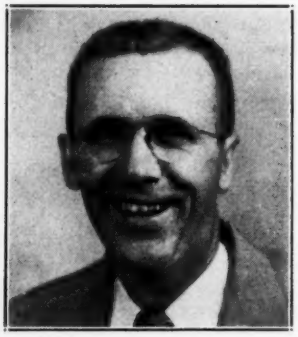
Born in Brockville, Ontario, Mr Fraser joined the Huntington (West Virginia) Works of The International Nickel Company, Inc, in May, 1923, serving in various technical and operating capacities before being promoted to the Company's New York Office in February, 1935, as assistant manager of the Production Department. He was elected vice president in March, 1947, and in the following June was made vice president in general charge of all plant operations in the United States of International Nickel. He was also an assistant vice president of the parent organization, The International Nickel Company of Canada, Limited, since June, 1943.

JOSEPH SEYER, SR

JOSEPH SEYER, SR, president of Seyer Silk Dyeing & Finishing Company, Haledon, N J, died on August 1st.

Mr Seyer was formerly president of the Silk & Rayon Printers & Dyers Association of America, Inc for five years and at the time of his death was chairman of the Board of Directors.

NAMES IN THE NEWS



W J Greene, Jr

R T VANDERBILT CO, INC, has named WILLIAM J GREENE, JR as technical service representative to the textile industry in New England, replacing MARSHALL W RAMSEY. Mr Greene previously had served as night superintendent at the Providence Dyeing, Bleaching and Calendering Company and manager of the Sumpter (S C) Textile Mills.

Mr Ramsey will make his headquarters in the Charlotte-Greenville area. Prior to his association with R T Vanderbilt, he was employed by the Philadelphia Quartermaster Depot as senior textile inspector.

M W Ramsey

D H Henderson holds a 20-pound muskellunge, caught on his recent trip to Wisconsin

INVALUABLE SERVICES performed by **Dr Cary R Wagner** as a top-ranking officer of General Aniline & Film Corporation were cited in an embossed set of resolutions recently presented by Company's Board of Directors on the occasion of Dr Wagner's resignation as senior vice president. Dr Wagner, who was elected Chairman of the Board's Executive Committee, resigned his operating post to be able to devote more time to his consulting business. He has been a Director of GAF for the past eight years and at one time was in charge of the Company's research program and later was Vice President, Operations. President **Jack Frye,** right, extends good wishes as he presents resolutions. Looking on are these Board members: **Thomas A Morgan, James A Purse, Jouett Shouse, Col T Colburn Davis,** and **Harold Church Paull.**

BIG fish in the coastal waters of the Atlantic or in fresh water appear to be the same to D H HENDERSON, technical superintendent of the finishing department of Dan River Mills, Danville, Va.

Early this year, he pulled in a 63-inch, 61½-pound wahoo, largest reported off the North Carolina coast in several years. Now he's added two of the biggest fresh water fish—the muskellunge—to his list.

On a recent trip to Wisconsin Headwaters Country (Vilas County), where he stayed at Y-Z Lodge, he hooked a 7 lb, 33-inch "keeper" on his first day out at Found Lake, using a black bucktail. On the second day, using the same lure, Mr Henderson came up with a 20-pounder, measuring 42 inches, out of Big St Germain Lake.

THE Board of Directors of the International Salt Company, Inc, at a meeting held on August 28th in New York, appointed as Vice Presidents EDSON K GREEN and MYRON L HYMAN.

Mr Green has been associated with International Salt since 1929, and has attained the position of general sales manager, which he continues to maintain.

Mr Hyman has been associated with International Salt since 1919, and has served in every phase of its sales organization. At present, he is general manager of sales for the Northern Division and Canada. He will continue to make his headquarters in Buffalo.

RUSSELL B NEWTON, president of Dan River Mills, Incorporated, has announced the promotion of W C HARRIS to the post of director of merchandising and sales with headquarters at the company's general sales office in New York.

Mr Harris previously was merchandise manager of the company's shirtings, chambrays, plain colored fabrics, storm wear and outer wear departments. He came to Dan River Mills a year ago from the Mooresville Mills, where he was vice president in charge of merchandising and sales and a member of the Board of Directors.

Mr Newton also announced the appointment of a Product Planning Committee whose function is to plan new product development, diversified styles and new fabric lines. This committee will be composed of Mr Harris; W J FULLERTON, merchandise manager of the dress goods department; B D BROWDER, vice president of manufacturing and L K FITZGERALD, director of research and development.

In addition, Messrs Harris and Fullerton have been added to the Management Policy Committee established some time ago. This committee also includes Mr Newton; Mr Browder; A B EMMERT, vice president cotton and raw material procurement; Mr Fitzgerald; R C GOURLEY, director of personnel administration; E J MACK, comptroller; and FRANK TALBOTT, JR, vice president and general counsel.

JAMES A RABBITT, Inco consulting engineer, and an authority on Japan and the Far East, retired from active service with The International Nickel Company, Inc, on August 31.

The services of Mr Rabbitt, who has been with Inco for more than 22 years, will continue to be available to the Company in an advisory capacity on Far Eastern affairs.

Mr Rabbitt, a native of Stamford, Connecticut, was 75 on August 6.

THE appointment of JOHN CROWTHER to the position of assistant sales manager has been announced by T A Haschke, sales manager, Stauffer Chemical Company.

Mr Crowther has been associated with Stauffer for seven years and was director of Eastern Division Research, the main offices of which are in Chauncey, N Y.

J Crowther

J Werner

DR JESSE WERNER has been named director of commercial development of the General Aniline Works Division of General Aniline & Film Corporation, where he will be in charge of all market research and development activities concerned with GAF's line of nondyestuff chemicals.

Dr Werner, who has been with General Aniline since 1938, previously served as technical assistant to John C Franklin, vice president in charge of operations. Prior to that he was assistant manager, Process Development Department of GAF's Grasselli, N J, plant.

TWO new Branch Managers have been named by the American Cyanamid Company's Coating Resins Department.

F S DUBBS, as Philadelphia Branch Manager, will be in charge of resin sales in that area. Prior to the new appointment, he served as assistant manager of the Industrial Chemical Division's Philadelphia Office.

J K MORRIS, as New England Branch Manager, will operate out of Cyanamid's newly-consolidated office in Boston, where he will continue to service the firm's coating resin accounts in the New England area. Mr Morris also transferred to the Coating Resins Department in 1949 from the Industrial Chemicals Division.

GEORGE J BRUYN, manager of Niagara Sales Office of Hooker Electrochemical Company, has been appointed Chief of the Alkalies Section, Inorganic and Agricultural Chemicals Branch of the Chemical Division, National Production Authority, effective August 1, 1952. The temporary appointment is on a WOC basis, and Mr Bruyn will be in Washington until April 1st, 1953. He has been granted a leave of absence by the Hooker Company for the tenure of the appointment. During World War II he worked closely with the War Production Board on allocation of Hooker chemicals required for military purposes.

T R Moore

TOM R MOORE has been appointed assistant to the vice-president and general sales manager of the General Dyestuff Corporation, New York. Mr Moore formerly was sales service manager for the Antara Chemicals Division of General Dyestuff.

Mr Moore started his career in the chemical industry in 1935 with The Sherwin-Williams Company. In 1941 he joined the National Carbon Company as a sales engineer and became associated with the Antara Division as sales engineer five years later. Mr Moore was appointed sales service manager in 1950.

W J Evert

WILLIAM J EVERT has assumed sales duties in the New York offices of Synthane Corporation (Oaks, Pa), manfacturers and fabricators of laminated plastics. Mr Evert is the second salesman to be added to the company's New York office in recent weeks. He reportedly will handle accounts in the Upper New York State, Brooklyn and Long Island areas.

J W ROSS has been promoted to Philadelphia District Manager for Carbide and Carbon Chemicals Co, a division of Union Carbide and Carbon Corp. Mr Ross, who joined the Company in 1936, came to Philadelphia from Atlanta in 1944. He moves up to fill the position formerly held by R M JOSLIN, JR. who has been appointed Midwestern Division Manager for the Company in Chicago.

G L Brown

GEORGE L BROWN has been elected vice-president in charge of manufacturing for the Caldwell Chemical Company, Inc, New York. This will be in addition to his duties as head of the company's Midwest operations, with offices in Detroit.

Mr Brown joined the Caldwell organization in 1950.

NEW assignments for four Cellulose Products Department district sales managers have been announced by Hercules Powder Company.

The new assignments are:

JAMES R YEAGER, manager lacquer promotion, Wilmington.

PHILIP F ROBB, district sales manager, New York City.

HENRY P GRACE, district sales manager, Detroit.

HOWARD S LAWTON, district sales manager, Cincinnati.

Mr Robb, who has been district sales manager in Detroit since 1936, joined Hercules in 1923. Mr Grace, who has been manager of the Cincinnati territory for the past five years, has been with the Company since 1940.

Mr Lawton has had experience in both the manufacturing and technical sales of cellulose derivatives. Mr Yeager began work at Hercules' Parlin, N J, plant in 1924. His experience is in all phases of the manufacture and sales of nitrocellulose and other cellulose derivatives for lacquer.

HARMON HOWORTH, manager of the Celanese Corporation of America plant at Rock Hill, South Carolina, since it came into production in 1948, has been promoted to a new position of greater responsibility in the recently formed Textile Division of the Company. He is moving to the company's New York offices.

ALEXANDER ROSE, assistant plant manager at Rock Hill, succeeds Mr Howorth as plant manager.

E Hart

AT A MEETING of the Board of Directors of Food Machinery and Chemical Corporation, JOSEPH B CARY was elected chairman of the Executive Committee and will share with President PAUL L DAVIES, responsibility for overall Corporate activities.

ERNEST HART, formerly a vice president of FMC and president of its Niagara Chemical Division was elected an executive vice president of the Corporation and will be responsible for supervising and coordinating the operations of all FMC Chemical Divisions. These now include Buffalo Electro-Chemical Division, Buffalo, N Y; Niagara Chemical Division, Middleport, N Y; Ohio-Apex Division, Nitro, W Va and Westvaco Chemical Division, New York, N Y. Mr Hart will make his headquarters in the Eastern Corporate Administrative Offices of Food Machinery and Chemical Corporation in the Chrysler Building East, New York.

Simultaneously, BENJAMIN C CARTER, formerly vice president and controller of FMC was elected an executive vice president of the Corporation and will be responsible for supervising and coordinating the Mechanical Divisions of FMC. Mr Carter's headquarters will be at the Corporate Administrative Offices in San Jose, California.

J C VERNON, previously vice president and sales manager of the Niagara Chemical Division succeeds Mr Hart as president and manager of that Division with headquarters at Middleport, N Y.

HENRY R MICHEL has been named director of purchases of Celanese Corporation of America. He assumes all the duties formerly performed by F W PAFFEN, whose full time is now required by procurement activities for Celanese Central, S A, recently established global organization of the company.

Mr Michel had been with Westinghouse Electric Corporation since 1920, and at the time of his resignation was assistant to the vice president in charge of purchasing. From 1938 to 1950 he was purchasing agent of the East Pittsburgh works of the company.

B L Hathorne

BL HATHORNE has joined the staff of Glyco Products Co, Inc in the capacity of assistant to the vice president in charge of development following his recent resignation as manager of the Auxiliary Products Division of Geigy Company. Prior to his affiliation with Geigy, Mr Hathorne was a consultant to the textile industry. He was formerly in charge of research and field development for the Tubize Chatillon Corporation.

In his new capacity, Mr Hathorne will devote full time to Glyco's expanding activities, with particular emphasis on the development of new products.

J V Stauf

JOHN V STAUF, formerly executive assistant to the president of the Solvay process division of Allied Chemical and Dye Corporation, has joined the staff of the Chemical Division of McKesson & Robbins, Inc.

Mr Stauf had been associated with Allied Chemical and Dye for the past 33 years, first with the general chemical division and then as vice president of the Solvay sales division. Last year he was made executive assistant to the president of the Solvay process division.

Mr Stauf will have his office in McKesson's New York headquarters, 155 East 44th Street, New York.

B B Bright

AMERICAN VISCOSE CORPORATION announces that B BROOKE BRIGHT has been appointed to head up its Technical and Professional Recruiting Division. In this capacity, Mr Bright will be in charge of recruiting technical and professional personnel, and his activities will be integrated with the Company's new executive development program.

In his new position, Mr Bright succeeds C STUART BROWN, who will hereafter devote all his time to public relations under HARRY L DALTON, vice-president in charge of sales and public relations.

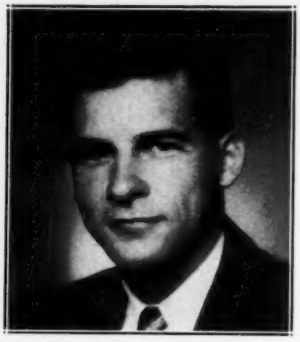
Mr Bright, formerly associated with the Atlantic Refining Company as personnel supervisor, will be located at the company's executive offices at 1617 Pennsylvania Boulevard, Philadelphia, Pennsylvania.

ERIC J MONAGHAN, one of the nation's foremost dye experts, retired last month after a 35-year career with the Du Pont Company.

Born in Madgeburg, Germany, the son of an attache at the American Embassy in Berlin, Mr Monaghan spent his early years in Germany.

In the spring of 1909, he joined the Badische Company in New York, as a colorist. When the submarine Deutschland broke through the English blockade from Germany to Baltimore during the first World War, Mr Monaghan did a major portion of the testing and standardizing of the cargo of dyes. He started with the Du Pont Company in 1917 as assistant director of what later became the Technical Laboratory at Deepwater Point, N J.

On January 1, 1934, he was transferred to Wilmington as assistant director of sales for the Dyestuffs Division, becoming director of domestic sales on August 1, 1946. He was named sales manager of the Dyestuffs Division in 1948. In the spring of this year, he was appointed assistant to the director of sales, Dyes and Chemicals Division, pending his retirement.



R H Roemer

ROBERT H ROEMER has been appointed general sales manager of Toscony Fabrics, Inc, where he has been employed since January, 1951 as manager of sales development. Previous to joining Toscony, he served 10 years with Carbide and Carbon Chemicals Company and, from 1945-1951, with Bakelite Company, both divisions of Union Carbide and Carbon Corp.

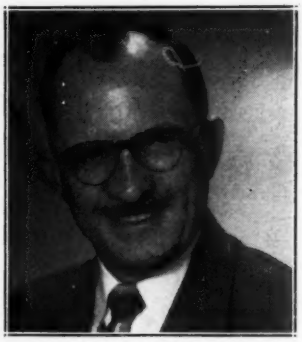
ANNOUNCEMENT of the appointment of RAYMOND E LAPEAN as managing director of Cyanamid Products Ltd was made on August 11. Mr Lapean, executive vice president of Chemical Construction Corporation since 1950, joined that organization in 1940 as a special assistant to the president.

HOOKER Electrochemical Company has announced the appointment of HORACE W HOOKER, JR as assistant to the general sales manager, PETER N MacLAREN as purchasing agent and ROBERT F SCHULTZ as assistant technical superintendent.

In connection with their management development program, the following temporary appointments have been made. JOSEPH A TARDIFF and JOHN D SWEENEY will serve as area supervisors, GEORGE E DUCKWALL as acting department head, and ALTON V THORPE as personnel supervisor in the Industrial Relations Department. These latter appointments represent the first steps in a more formal type of management development program and are part of an overall plan for developing reserve management potential to provide for the company's growth and potential.

JACK TURER, head of the analytical and textile service departments at Virginia-Carolina Chemical Corporation (Fiber Division) has joined the research staff of L Sonneborn Sons, Inc, New York, N Y, where he will concentrate on development work relating to the processing of synthetic fibers.

Mr Turer was research chemist for the U S Department of Agriculture before joining the Virginia-Carolina Chemical Corporation in 1945 to head up its research on synthetic protein fibers.



E O Smith

AS PART of its expansion program, the International Ticket Company, Newark, N J, has appointed EVAN O SMITH divisional manager of the Vat Dye Label Department.

While working as a chemist and colorist for Du Pont, Mr Smith realized the need for a fast label for washable fabrics and the possibilities of using vat dyes on ribbon. As a result, he has been a primary actuating influence in the development of the 100% color-fast printed label.

DR H G JOHNSTON heads the research development unit, production and marketing division, National Cotton Council, as of September 1. Mr Johnston has been head of the Department of Entomology at Texas A & M College since 1946.

NEW PRODUCTS AND DEVELOPMENTS

• Du Pont Introduces New Pressure Fabric Dyeing Machine

A major change in batch dyeing methods for fabrics is anticipated with the introduction in Wilmington, Del, last month of a new pressure fabric dyeing machine.

Although still in the development stage, the machine was demonstrated to more than 150 representatives of the nation's textile industry at a conference under the auspices of two of Du Pont's departments —the Textile Fibers Department and the Organic Chemicals Department.

The pressure dyeing machine is being developed by the Textile Research Division at the Newport Textile Laboratory of the Du Pont Company as part of the long-range research program to facilitate and perfect the dyeing of new synthetic-fiber fabrics, particularly those of Orlon acrylic fiber and Dacron polyester fiber.

Paul M Cole, senior research engineer of the Engineering Department's Machine Development Section, who designed the machine, pointed out that the development is not near completion but he said it potentially meets every one of the 14 characteristics of an "ideal dyeing machine".

"It needs the refinement of the textile machinery manufacturer and the experienced touch of the practical dyer," he said. He disclosed that the development is named "Barotor", because its principal mechanical parts consist of a rotor and bars within a steel cylinder. The machine is normally operated at 250° F, corresponding to a pressure of 15 psi.

Du Pont developed the "Barotor" largely as a service to customers, Mr Cole stated, and licensing arrangements under pending patents are being made with several textile machinery firms. Dyers will reportedly be free to use the "Barotor" without royalty.

The 14 points of the ideal dyeing machine which the "Barotor" potentially mee.s, according to Mr Cole, are:

1) Fabric is open width at all times
2) Tensionless and wrinkle free
3) All points in uniform and frequent bath contact
4) Operability with all fabric constructions, both filament and spun
5) Handling of batches as small as 100 yards and batches in excess of 1000 yards
6) Rapid bath agitation or circulation
7) Wide range of bath ratios permissible
8) Exact duplication of conditions between laboratory and production scale
9) No need for attention during dyeing
10) Fabric sampling and bath additions made without opening vessel or losing pressure
11) Samples always representative of entire fabric
12) Essentially cylindrical in shape for 100% utilization of the lowest cost pressure vessel
13) Minimum cost of operation and maintenance
14) Investment commensurate with productivity in comparison with atmospheric dyeing of natural fibers

The announcement of the Barotor closed a full day conference on dyeing Orlon acrylic fiber and Dacron polyester fiber. The program, under the direction of Dr J H Trepagnier of the Textile Fibers Department, included papers on the dyeing of Orlon, by Dr R H Blaker, Dr P L Meunier and G T Hug of the Organic Chemicals Department. The dyeing of Dacron was discussed in papers delivered by Dr W R Remington of Jackson Laboratory, Dr R J Andres of Textile Fibers Research and Dr Meunier.

In a general session preceding questions and answers in the afternoon, Dr J E Smith of the Organic Chemicals Department told of new Du Pont antistatic agents and W G Rhoads, Textile Fibers Department, spoke on advantages of aqueous dyeing at temperatures above 212° F.

• Hartofume C

The Hart Products Corp, 1440 Broadway, New York, N Y, has released information on Hartofume C, their new gas fading inhibitor in liquid form. It is said to be a permanent antigas fader which provides a complete range of gas fading protection for dyed acetate fabrics. This protection is said to be unaffected by washing, dry cleaning or stripping.

Hartofume C is claimed to be substantive to cellulose acetate fibers and, therefore, may be applied to the cloth directly in the dyebath. The manufacturer reports the following additional advantages:

(a) disperses readily in water to give stable, milky emulsions,

(b) needs no additional solubilizers for dispersion in water,

(c) may be applied in jigs, dye boxes, circulating liquor machines and by padding,

(d) is not removed from the cloth by stripping agents (in redyeing, no additional Hartofume C is required),

(e) does not affect the light fastness of most acetate and direct dyes, and

(f) does not affect the shades of dyestuffs (no allowance need be made in matching shades).

• Pure Orthodichlorobenzene

A new development in the field of chlorine chemistry which may improve the synthesis of dyestuffs, intermediates, pharmaceuticals and rubber chemicals was announced last month by D H Ross, director of product development of the Solvay Process Division, Allied Chemical & Dye Corporation.

Solvay has reportedly perfected a pilot plant technique for the manufacture of an extremely pure orthodichlorobenzene, having less than ½% total impurities which, it is believed, will prove of great value to manufacturers of various organic chemicals. The use of this material in the production of dyes is expected to provide greater uniformity of shade from batch to batch. Solvay has stated that, if a substantial market is developed for the new product as the result of experimentation by companies taking samples of the high-purity orthodichlorobenzene, they will install facilities for its manufacture in quantities to meet the demand.

"Pure ortho" is said to be prepared under carefully controlled conditions in equipment which allows the dielectric constant and other physical properties influenced by the presence of contaminating impurities to be held within rigid limits.

In certain applications commercial grades of orthodichlorobenzene leave undesirable residual odors due to high boiling components such as trichlorobenzenes. These undesirable constituents are said to be completely removed from "pure ortho", a faster evaporating product.

Samples and further technical information may be obtained by writing the Product Development Department, Solvay Process Division, Allied Chemical & Dye Corporation, 40 Rector Street, New York 6, N Y.

• Nonic 261

Sharples Chemicals, Inc, Philadelphia, Pa, announces the availability of a new member of its Nonic family of surface-active agents—Nonic 261. The Nonics are polyethylene glycol tert-dodecylthioethers, nonionic surface-active agents.

Nonic 261, a liquid, is said to be an excellent wetting agent and an effective emulsifier for animal, fish and mineral oils.

It is suggested by the manufacturer for applications in the textile and other industries as an emulsifier and as a degreaser for metal parts.

• New Interchemical Line of Fast-to-Light Direct Dyes

For dyeing cotton and rayon, Interchemical Corporation, Textile Colors Division, Hawthorne, N J, has introduced a new line of fast-to-light direct dyes—the Arilite Dyes, which are said to possess excellent penetrating and leveling properties. Because they have no affinity for acetate, they are useful for cross-dyeing rayon-acetate blends, it is pointed out. Fabrics dyed with Arilite Dyes reportedly require no special aftertreatment.

Arilite Dyes designated by the letter "U" are said to be particularly valuable for dyeing fabrics which are to be treated with resin finishes. Shades produced with these dyes exhibit minimum change when treated with resin finishes, it is claimed, and lightfastness before and after resin treatment is outstanding. Washfastness after resin treatment is reportedly improved.

• Dextrol Rayon Crepe Oil #114

A new oil for crepe soaking of rayon skeins or cakes has been developed by Dexter Chemical Corporation, New York, (Dextrol Rayon Crepe Oil #114). A blend of sulfonated oils with free cocoanut oil, it is reported to form a very stable emulsion for use in either machine or tub soaking.

TECHNICAL LITERATURE

Brochure on Wool Textile Shrinkage

Wool Bureau Incorporated
16 West 46th Street
New York 19, N Y

A brochure on the cause and treatment of various types of shrinkage in wool textiles, released on August 17 by The Wool Bureau, Inc, contains information of primary interest to the consumer and educator on types of wool shrinkage and treatments available.

Among the features of the brochure are a discussion of new theories on the phenomenon of felting shrinkage, a listing of characteristics for each of the commercial processes or combatting shrinkage, i e, wet chlorination, resin treatment and latex treatments, and a list of trade marks of firms using these processes.

Twitchell Worsted Oil Booklet

Emery Industries, Inc
Dept 5, Carew Tower
Cincinnati 2, Ohio
Available on request

Properties of the new high-quality Twitchell 7421 Worsted Oil for static control, such as antistatic, emulsion fluidity, emulsion stability, resistance to yellowing and odor development, scourability, and rust preventive ability, are fully discussed.

n-Butyl Alcohol

Bulletin F-7909
Carbide and Carbon Chemicals Co
30 East 42nd Street
New York 17, N Y
Available on request

F-7909 contains material on chemical, physical and physiological properties, solubilities, shipping data, and applications of this colorless, mild-odored liquid, which finds a variety of uses as a solvent, intermediate or diluent.

Technical Bulletin on Aquex BNWC

Aquex Development & Sales Corp
Whippany, New Jersey
Available on request

The use of this new cationic resin for textile finishing is outlined in this technical bulletin, which describes a process for the attainment of dimensional stability on 100% spun viscose and viscose rayon blend shirting and dress fabrics. The process is said to give low residual shrinkage, with no chlorine retention or progressive shrinkage on extended washing, while improved wet strength and ease in producing different types of hand are brought about.

X-2——A New Chemical for the Modification of Cellulose

Chemical Development Division
Dan River Mills, Inc
Danville, Virginia

New data on the properties of "X-2" rayon fabrics and on the application of the "X-2" chemical to viscose rayon fabrics in the textile finishing plant are disclosed in this technical service bulletin which has just been issued.

Limited quantities of "X-2" rayon fabrics have been produced by Dan River Mills and are now in the market, particularly in the men's shirting field, it is reported. Fairly complete lines are expected to be made available in the near future. These fabrics are also being produced by other mills which completed arrangements with Dan River Mills for the utilization of the X-2 chemical sometime ago.

An introductory statement to the X-2 service bulletin clarifies some of the basic facts relative to this chemical treatment, as follows:

"More than ten years of continuous research by the Dan River laboratories in the application of finishes to cotton, rayon and rayon blends resulted in the development of the X-2 chemical.

"This research covered all the known finishes now in the market and hundreds of others of Dan River's own origination. X-2 is a Dan River origination.

"Stated literally, X-2 is a readily applied chemical treatment that permanently stabilizes rayon and rayon-blend fabrics.

"The modification of the molecular structure of the fibers in an X-2 treated rayon or rayon blend fabric produces changes in the properties of the fiber which are an integral part of the fiber and cannot be altered or removed by washing or wear, or by any method short of actual destruction of the fiber itself.

"X-2 is a chemical that is entirely new to the industry. It is not a resin, nor is it related to any of the resins. Further, it is not in any way comparable to any of the resins, either in chemical composition, or in the permanent end-results when applied to rayon and rayon blend fabrics.

"The application of X-2 requires no equipment other than that now in use in mills and finishing plants for the application of resin finishes."

The bulletin goes into rather precise descriptions of the properties contributed to rayon and rayon blend fabrics by the X-2 chemical and places particular emphasis on such features as increased wear-life, complete and permanent launderability, almost complete elimination of shrinking and stretching, and the absence of any chlorine retention.

The second part of the bulletin is devoted to the X-2 chemical and its application, with a preliminary statement on the chemical action that results when rayon and rayon blend fabrics are treated, and how this chemical action supplements the molecular structure of the fiber and thus permanently establishes the new and desirable properties.

Methods of application, such as avoidance of tensions, souring, padding, drying, and curing are referred to in some detail. The method by which the hand of the finished fabric may be varied is described.

The use of the X-2 chemical is being extended to other cellulosic fibers with encouraging results, it is pointed out. Acetate fabrics treated with X-2 are said to show outstanding properties and present development work reportedly indicates that a modified form of X-2 for cotton will give the same outstanding properties to cotton fabrics that the present X-2 gives to rayon.

New Honeywell Literature

Minneapolis-Honeywell Regulator Co
Brown Instruments Division
Station 40
Wayne and Windrim Avenues
Philadelphia 44, Pa
Available on request

REISSUE OF BULLETIN 15-14 "INSTRUMENTS ACCELERATE RESEARCH"

Bulletin 15-14, "Instruments Accelerate Research", has been augmented to include many items not covered in the previous edition and has been brought up to date to include the latest designs of various analytical equipment. The bulletin now comprises ninety-six pages and has complete illustrations with pictures of the latest instrument and equipment in the laboratory, analytical and measurement fields.

ELECTRONIK PRECISION INDICATING POTENTIOMETERS

Honeywell's specification sheet 177 describes and illustrates both the new console type operator's desk and the Brown *ElectroniK* precision indicator. It describes the precision indicator for both single and multipoint indication of variables as well as the extended range indicator and shows how this indicator, when combined with the operator's desk and banks of switches, permits the operator to review and log many variable readings while sitting at the desk.

INDUSTRIAL PROCESS CONTROL

39-page Second Quarter 1952 issue of "Instrumentation" includes informative technological articles on Program Control, Time Proportioning Control and Graphic Panels, and also interesting descriptions of ways in which major companies apply instrumentation in the successful measurement and control of important process variables.

POTTER-BROWN ELECTRONIC FLOW METER

Instrumentation Data Sheet 10.3-5 describes the Potter-Brown Flowmeter "for measurment of fluids over an extremely wide range of temperatures and pressures with an accuracy of ½%." The new system is said to be easily installed, has linear characteristics, and can be used to measure, record and control flow at one or more points.

The Potter-Brown meter can reportedly be used to measure the flow of chemical compounds including strong caustics and fuming nitric acid.

ELECTRONIK STRIP CHART PROGRAM CONTROLLERS

Bulletin 1501 describes *ElectroniK* Strip Chart Program Controllers with Electric Set Point Index Drive, used for automatic regulation of time-temperature control programs.

Models available, construction details, typical applications, specifications, dimensions and data on auxiliary equipment is included.

INSTRUMENTATION—THIRD QUARTER 1952

A description of the ultra-modern graphic control panel for the new White House air-conditioning system, is one of the features in the 40-page Third Quarter 1952 issue of "Instrumentation".

Also included are an informative article on *Teflon* valve packing; one of the instrument courses at Case Institute of Technology; and plant stories that tell how companies such as Merck & Co, Savannah Electric and Power Company, Atlas Powder Company, and others make use of engineered instrumentation to keep production and quality up—costs down.

Industrial Insulations

Baldwin-Hill Company
1086 Breunig Avenue
Trenton, New Jersey
Available on request

This 20-page illustrated catalog describes insulating materials which cover the complete temperature range from-150° F to 1800° F. Insulating cement, block, blanket, felt, and pipe covering are a few of the products shown in the catalog.

Complete with thermal-conductivity graphs and heat loss charts, the catalog also shows list prices subject to trade discounts. Brief application descriptions together with typical uses, sizes, packaging, and densities are also included.

Dyeing and Wet Finishing Techniques for Dynel (1952 Edition)

Textile Fibers Department
Carbide and Carbon Chemicals Company
Union Carbide and Carbon Corporation
30 East 42nd Street
New York 17, N Y
Available on request

Techniques discussed in this manual bring the recommended procedures up to date with new developments and experience gained since the March, 1951, manual was published. It covers new methods for dyeing Dynel with mixtures of acetate and acid dyes and an improved process for dyeing by means of the "controlled addition of copper" technique. Sections on stock and package dyeing have been revised and a section on top dyeing added.

Other sections discuss basic, direct, resist and soluble vat colors, bleaching and finishing.

Pulverizers for Grindable and Ungrindable Materials

Mead Mill Company
14620 East Seven Mile Rd
Detroit 5, Mich
Available on request

The complete line of Mead Mill high speed impact type pulverizers are covered in this four-page catalog. Typical grindable and ungrindable materials pulverized by Mead Mills are listed on the cover. Detailed drawings give the dimensions of all five mills that range in horsepower from 1 to 30. A large cutaway view graphically shows the impact principle employed by all Mead Pulverizers. Typical installation drawings are also included. A graph is featured that gives typical production information for tough resilient material, friable and highly friable materials.

Shown on the back cover is the new mobile laboratory pulverizer designed for convenience in successively processing different materials.

Hydrocide S-X Colorless

Building Products Division
L Sonneborn Sons, Inc
New York 11, N Y

Technical Data Guide No. 40 discusses Hydrocide S-X Colorless, sonneborn's new invisible water-repellent Silicone, which gives water-repellency in depth to exterior concrete and masonry walls. Features of the bulletin include a performance comparison with competitive products, a summary of performance data, methods of use, specifications, and packaging and shipping data.

"Albone" 35 Hydrogen Peroxide Properties and Applications

Electrochemicals Department
E I du Pont de Nemours & Co, Inc
Wilmington 98, Delaware
Available on request

This eight-page bulletin lists chemical and physical properties, describes control of reactions and decomposition, methods of analysis, and gives packaging information. It also lists construction materials for alkaline and acid peroxide solutions and includes a section on uses covering bleaching of the principal textile fibers, leather, fur, etc. Additional technical bulletins by Du Pont on hydrogen peroxide are listed at the end.

Gurley Bulletin 1400

W & L E Gurley
Troy, New York
Available on request

Methods of testing paper, paperboard, textiles, plastics and other sheet materials, as well as the complete line of Gurley Instruments for performing the tests, are described in this new bulletin by a 107-year-old manufacturer of scientific instruments.

A new feature of the bulletin is a table of "Air Resistance Equivalents", which is designed to help the technician select the type of instrument best adapted to various materials.

Included in the 20-page booklet is information on the following instruments and accessories: Curley Stiffness Tester (Motor Operated); Precision Strip Cutter; Curley-Hill S-P-S (Smoothness-Porosity-Softness) Tester; Permeometers; Gurley-Cobb Sizing Tester; Densometers; Densometer Gaskets and Automatic Timing Attachments.

Pressure-Type Filters for the Removal of Visible Suspended Matter From Water

Bulletin WC-107
Graver Water Conditioning Co
Department 41-7
216 West 14th Street
New York 11, N Y
Available on request

Bulletin WC-107 describes the details of design and construction of these pressure-type filters, which are built in both vertical and horizontal types, together with all the accessory equipment. The various available arrangements of valves and piping and aids in their selection for individual requirements are also fully discussed. Illustrations of installations are included, together with complete tables of capacities, dimensions and weights for both horizontal and vertical filters.

Laboratory Planning Kit

Fisher Scientific Co
717 Forbes Street
Pittsburgh, Pa
Available on request

Fisher Scientific Company has introduced this device as a means of suggesting methods of planning layout and use of their Unitized laboratory furniture.

Contents include a catalog of the laboratory furniture, photos of lab installations showing various arrangements, "how to use" instructions, dimensions, furniture scaled cutouts and graph paper.

Magnetic Separators for the Textile Industry

Eriez Manufacturing Company
Erie, Pennsylvania
Available on request

A new, six-page, two-color brochure (No. B-563) explains how magnetic separators provide a simple, economical, efficient means of removing iron and steel trash from wet or dry textile fibers, thereby preventing spark-caused fires and protecting machinery.

Eriez' brochure describes with data and illustrations typical magnet installations in the processing of cotton, waste, wool, and wool-rayon blends. Also included are dimensions and specifications for Eriez Plate ATOMagnets, ATOMagnets Pulleys, Pneumatic Assemblies (Humps), and Magnetic Traps developed for the textile industry.

The new Hyflux Alnico V used in Eriez separators is said to give the greatest magnetic strength of any material in commercial use. No outside power or electrical connections are required since all units are non-electric and complete for installation on wool or steel. According to the Factory Insurance Association, "numerous installations of permanent magnets are operating with complete satisfaction in many textile mills." All Eriez magnetic separators developed for textile use reportedly bear the Associated Factory Mutual Stamp of Approval.

Modeling Molecules

Fisher Scientific Co
717 Forbes Street
Pittsburgh, Pa
Available on request

A reprint of the lead article by Dr Douglas G Nicholson in *Chemical and Engineering News 30,* 3164 (August 4, 1952). 4 pp, illustrated.

This is reputedly is the first complete history and analysis of atom models.

Dr Nicholoson's survey includes commercially available atom models as well as those devised by university staffs throughout the United States.

• GDC Technical Circulars

Summer releases of technical literature of the General Dyestuff Corporation, 435 Hudson St, New York 14, N Y, include the following circulars:

G-709—Naphtol AS-DB
G-714—Fastusol Blue LFGL
G-715—Fastusol Orange RRL-CF
G-716—Celliton Fast Blue FFRN Extra Conc
G-717—Celliton Fast Navy Blue BA-CF
G-718—Celliton Fast Red Violet RNA-CF

Naphtol AS-DB is a substantive Naphtol of the AS series, which is used on cotton and rayon yarns and piece goods for dyeing brown shades with mostly good to excellent fastness properties by coupling with fast color salts or diazotized fast color bases, it is stated. G-709 is devoted to a discussion of fastness properties and uses, dyeing properties, application (preparation of material), naphtholation (dissolving methods, exhaustion and padding), development, soaping, and typical procedures in padding, in the jig, in the circulating machine, and application from a long bath. In addition to presenting a discharge formula as well as a table of fastness ratings, the circular includes three pages of sample dyeings on cotton yarn and mercerized cotton sheeting.

Fastusol Blue LFGL is a new straight direct dyestuff developed for dyeing fast-to-light blue and combination shades on cotton and rayon. An outstanding feature of this blue is its suitability for use on rayon piece goods that are to be given an anticrease resin finish as the shade and light fastness of dyeings on rayon are said to be scarcely affected by resin finishing. Other important features include dischargeability and suitability for improvement of wet fastness with Solidogen WF Pdr and copper salt. G-714 discusses, among other properties, shade and fastness, aftertreatment, anticrease-resin finishing, dischargeability, dyeing behavior. Other sections are devoted to uses, application, optional aftertreatment (batch method and with resin finish), and fastness of full dyeings on cotton and rayon. Mounted samples portray the product's effectiveness on cotton sheeting, rayon crepe and cotton-wool unions.

Fastusol Orange RRL-CF is a new straight direct dyestuff developed for dyeing fast-to-light oranges of good tinctorial value on cotton and rayon. It is said to be especially important for dyeing rayon piece goods that receive an anticrease resin finish because it is almost unique among oranges—particularly reddish oranges—in that its fastness to light is scarcely impaired by such aftertreatment. Under a section on "Properties", G-715 discusses shade, fastness, dischargeability, anticrease-resin finishing and dyeing properties. Uses (including rubberizing) and application of Fastusol Orange RRL-CF are outlined. Other features include fastness and property tables and sample dyeings.

Celliton Fast Blue FFRN Extra Conc, Celliton Fast Navy Blue BA-CF, and Celliton Fast Red Violet RNA-CF are discussed, respectively, in G-716, G-717 and G-718. All three releases include material on the relative applications to acetate and nylon, while the latter two also discuss rubberizing. Samples, fastness ratings and properties for each product are included.

According to GDC, Celliton Fast Blue FFRN Extra Conc is a straight, readily-dispersible anthraquinone dyestuff, which dyes acetate and also nylon directly to give very bright, fast-to-light blues. Celliton Fast Navy Blue BA-CF is also characterized as a straight, readily-dispersible, direct-dyeing dyestuff (Pr 232) for cellulose acetate and also for nylon, which is said to give deep navy shades of better than average fastness to light. Little change of shade in artificial light is reported. Celliton Fast Red Violet RNA-CF, a straight anthraquinone type (Pr 237), is for direct dyeing of very bright red-violet shades. Light fastness is reported excellent, and tinctorial value very good on cellulose acetate. GDC also suggests its use for equally bright violet shades of good light fastness on nylon.

• Six Russian Chemical Journals Now in English Translation

In response to demand for English translations of Russian scientific papers, four additional Soviet chemical journals will be published in complete English translation, beginning with the 1952 volumes, by Consultants Bureau, 152 West 42nd Street, New York 18, N Y, as follows:

The Journal of Analytical Chemistry of the USSR, $80.00 per year; *Bulletin of the Academy of Sciences of the USSR, Division of Chemical Science*, $80.00 per year; *Colloid Journal*, $80.00 per year; and *Progress of Chemistry*, $95.00 per year.

These four journals, all publications of the Academy of Sciences of the USSR, were removed from the list of Soviet technical journals available for export in 1952, and hence are not available in the original Russian on a subscription basis. These USSR export restrictions will not affect the Consultants Bureau program of translating entire Soviet chemical journals, according to the publisher.

Individual issues may be purchased at from $10 to $20, depending on the journal, and single papers may be purchased at $7.50, regardless of length.

• Cyanamid Booklet on Acrylonitrile Uses

Copies of a booklet which describes numerous uses of acrylonitrile in making resin adhesives may be obtained on request by writing to the Synthetic Organic Chemicals Department, American Cyanamid Company, 30 Rockefeller Plaza, New York 20, N Y.

• Stauffer Catalog Revised

Stauffer Chemical Company has issued a revised 112-page edition of their general catalog covering basic chemicals for industry and agriculture. As in the earlier editions, considerable data are given on all products, and numerous tables on specific gravity, viscosity, specific heat, solubility and other properties give assistance to the user of Stauffer chemicals. For copies, write the company at 420 Lexington Ave, New York 17, N Y or its nearest branch office.

Barnstead Purity Meter

Bulletin 125
Barnstead Still & Sterilizer Co
181 Lanesville Terrace
Forest Hills, Boston 31, Mass
Available on request

Details of the new Barnstead Purity Meter, an electronic testing device for determining the purity of either distilled or demineralized water, are covered in this bulletin.

Operating on the principle that the more impurities in the water, the greater its electrical conductivity, this instrument reportedly gives readings directly in parts-per-million. A conversion table, mounted on the side, shows the equivalent resistance and conductance.

A section of the bulletin is devoted to the Barnstead Conductivity Diverter, an automatic control device. Operated by a conductivity cell in the pure water line, this special controller-type purity meter is designed to permit only water of predetermined purity to enter the storage tank.

The Barnstead Purity Meter is available with either platinum dipped, glass dip-cell or nickel pointed, hard rubber dip-cell.

Dow Booklet on Caustic Soda

Alkali Sales Department
The Dow Chemical Company
Midland, Michigan
Available on request

The newly-revised booklet specifically deals with the following:

1) The Manufacture of Caustic Soda
2) Chemical and Physical Properties
3) Economics Involved
4) Methods of Handling and Storing
5) Technical Data
6) Methods of Analysis
7) First Aid Treatment for Caustic Burns

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Individual issues may be purchased at from $10 to $20, depending on the journal, and single papers may be purchased at $7.50, regardless of length.

• Cyanamid Booklet on Acrylonitrile Uses

Copies of a booklet which describes numerous uses of acrylonitrile in making resin adhesives may be obtained on request by writing to the Synthetic Organic Chemicals Department, American Cyanamid Company, 30 Rockefeller Plaza, New York 20, N Y.

• Stauffer Catalog Revised

Stauffer Chemical Company has issued a revised 112-page edition of their general catalog covering basic chemicals for industry and agriculture. As in the earlier editions, considerable data are given on all products, and numerous tables on specific gravity, viscosity, specific heat, solubility and other properties give assistance to the user of Stauffer chemicals. For copies, write the company at 420 Lexington Ave, New York 17, N Y or its nearest branch office.

Barnstead Purity Meter

Bulletin 125
Barnstead Still & Sterilizer Co
181 Lanesville Terrace
Forest Hills, Boston 31, Mass
Available on request

Details of the new Barnstead Purity Meter, an electronic testing device for determining the purity of either distilled or demineralized water, are covered in this bulletin.

Operating on the principle that the more impurities in the water, the greater its electrical conductivity, this instrument reportedly gives readings directly in parts-per-million. A conversion table, mounted on the side, shows the equivalent resistance and conductance.

A section of the bulletin is devoted to the Barnstead Conductivity Diverter, an automatic control device. Operated by a conductivity cell in the pure water line, this special controller-type purity meter is designed to permit only water of predetermined purity to enter the storage tank.

The Barnstead Purity Meter is available with either platinum dipped, glass dip-cell or nickel pointed, hard rubber dip-cell.

Dow Booklet on Caustic Soda

Alkali Sales Department
The Dow Chemical Company
Midland, Michigan
Available on request

The newly-revised booklet specifically deals with the following:

1) The Manufacture of Caustic Soda
2) Chemical and Physical Properties
3) Economics Involved
4) Methods of Handling and Storing
5) Technical Data
6) Methods of Analysis
7) First Aid Treatment for Caustic Burns